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Goddard Space Flight Center

1971 NASA/Goddard-Aerospace Industry  
Battery Workshop

Second Day

Greenbelt, Maryland  
18 November 1971

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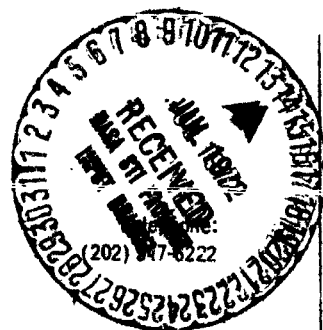
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## NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Goddard Space Flight Center

Greenbelt, Maryland

## 1971 NASA Goddard-Aerospace Industry

Battery Workshop

(Continued)

Chairman: Gerald Halpert

Room 231, Building 7,  
Goddard Space Flight Center,  
Greenbelt, Maryland.

Thursday, 18 November 1971.

The meeting was called to order at 9:15 a.m.,  
pursuant to adjournment.

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P R O C E E D I N G S

HALPERT: Gentlemen, can we take our seats, please?

Good morning. Welcome, all, back to the third session on the nickel-cadmium batteries, the NASA/Goddard Battery Workshop.

If you have not signed the sign-in sheet, if you were not here yesterday or if you were here and did not sign the sheet, we would appreciate you signing it to make sure that we have recorded all the people who are here for this meeting.

At the sides of the room we have some additional material. Bill Webster brought over three samples of 100 ampere hour cells from Eagle Picher, GE, and Gulton for you to inspect during the break time if you have a free moment. Their weights are listed.

Tom Hennigan would like to say a couple of words before we get going with our materials section.

HENNIGAN: Recently at these meetings we've been

NV-22

1 talking more and more about the company's products and identify-  
2 ing them, which is good. But I noticed yesterday there has to be  
3 some clarification on some of these things: did you treat  
4 them or did you do something to them, because this is not  
5 really the same product any more if you did something.

6 I guess in the separator area this becomes parti-  
7 cularly touchy because as far as I know, all fibers have a  
8 lubricant on the surface so that fibers can be handled in the  
9 machinery. It just so happens that lubricants are usually good  
10 wetting agents and if you remove them before you put it into  
11 a cell, you're going to change the characteristics of that  
12 material and no longer can you identify it by the manufacturer's  
13 number unless you put some kind of a subscript after it like  
14 I used on mine, "AR" for "as received," "W" for "washed," and  
15 "T" for "treated."

16 I couldn't go into all the things we did but at  
17 least you knew we had changed it. To go into all the treat-  
18 ments and washing would take quite a long time.

19 I think we have to do this to protect those who  
20 gave us or sold us the material.

21 Thank you.

22 HALPERT: If anyone would like to make corrections  
23 in their parts of the paper, if they'll contact me within the  
24 next week I'll be glad to insert the proper subscripts so that  
25 it is recorded properly.

MV-3

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For our first speaker this morning on materials, Bob Howard from Eagle Picher is going to talk about machine processing for dry sintered plaque.

Bob Howard.

HOWARD: The purpose of this paper is to present some of the work accomplished on the NASA process variable study, contract number NAS 521159. This presentation pertains to the development of the semi-automatic process for the dry sintering of porous nickel plaques.

The authors of this paper, Mr. Walt McCarter, Dr. Don Crabtree, and myself, wish to acknowledge the technical assistance of Mr. Floyd Ford, Goddard Space Flight Center.

The design concept basic to the program was to develop a machine system to manufacture dry sintered nickel plaques. The target specifications were plaque thickness plus or minus 1/1,000 of an inch anywhere on a plaque and from plaque to plaque; plaque porosity of .4 of one percent anywhere on a plaque and from plaque to plaque. Yield weights were 85 percent of all plaques produced were within plus or minus 2 grams on a 100-square-inch plaque area.

eb4

1 The design concept dictated close machine toler-  
2 ances, machine stability, reproducibility of machine opera-  
3 tions, and no hand labor which would directly influence the  
4 forming of the nickel plaque.

5 A machine system which satisfactorily met all of  
6 the foregoing criteria evolved during the development program.  
7 Figure 1 presents a photograph of the final configuration of  
8 the plaque-forming machine.

9 Floyd, would you turn the lights down, please?

10 (Slide 109.)

11 To the right beneath the control panel is shown  
12 a typical carrier platen which is used in conjunction with  
13 the machine. The platen is approximately 1 foot square and  
14 is fabricated from 031 thick nickel 200 sheet.

15 Located in the center of the platen is a nickel  
16 substrate screen. Prior to joining the screen to the platen,  
17 the platen is flattened in an impact press with an automatic  
18 feed. The screen is processed through a neophrene-faced  
19 press during which a series of 60 each, 1/8th inch diameter  
20 embossed studs are formed in the screen.

21 The embossings hold the screen approximately  
22 7/1000ths above the platen thus assisting in positioning the  
23 screen in the center of the plaque.

24 The platen with the screen is inserted into the  
25 machine underneath the two parallel bars and a ground steel

eb5

1 table possessing a circumferential O-ring is activated upward  
2 and firmly holds the carrier platen against the underneath side  
3 of the parallel bars.

4 A vacuum is pulled over the entire lower side of  
5 the carrier platen. This vacuum flattens the platen against  
6 the vacuum table.

7 Next, the storage hopper containing the nickel  
8 powder lowers and a pre-determined quantity of nickel powder  
9 is automatically dispensed at the upper end of the platen.  
10 The hopper automatically retracts and there is a narrow pile  
11 of nickel powder immediately in front of the crossbars shown  
12 in Figure 1.

13 This crossbar is mounted at each end to bushing  
14 blocks which are carried on precision-ground shafting. The  
15 shafting is protected by shaft boots and each shaft is posi-  
16 tioned on an adjustable weigh mount.

17 The shafts, crossbar, parallel bars, vacuum table,  
18 and carrier platen are thus located precisely to one another,  
19 insuring the required dimensional control.

20 Carried beneath the crossbar are two semicircular  
21 polished rods mounted parallel to one another. The rear bar  
22 determines the final thickness of the unsintered plaque.  
23 Upon command of the machine operator, the crossbar assembly is  
24 actuated forward. This motion pushes the nickel powder across  
25 the platen and screen forming the unsintered plaque.

eb6<sup>1</sup>

1 After the plaque is formed, the vacuum table  
2 lowers, the operator removes the carrier platen and places it  
3 on a belt of the sintering furnace.

4 The crossbar is returned to the rear position and  
5 the operator removes any nickel powder from the vacuum table.

6 The machine is controlled employing a pneumatic  
7 logic system which is interlocked with various mechanical  
8 motions of the machine. The machine is housed in a temperature  
9 controlled room adjacent to the sintering furnace.

10 Several problems were incurred during the develop-  
11 ment, some of which deserve comment.

12 Ground ceramic plates were tried as carrier  
13 platens. These ceramic plates lead to an excellent quality  
14 plaque but repeated passes through the sintering furnace  
15 resulted in grain growth and eventual fracture. The ultimate  
16 solution was the nickel plate used in conjunction with the  
17 vacuum table.

18 Dimensional control on the nickel carrier platens  
19 is as good as that determined in early use of the ceramic  
20 platens.

21 The first method of distributing the nickel powder  
22 was to sift it onto the carrier platen and substrate screen.  
23 This procedure resulted in porosity variations as high as  
24 plus or minus 2 percent and in non-reproduceable weight of  
25 nickel powder laid down per unit area.

eb7

1 The present method of drawing a pile of nickel  
2 powder across the substrate screen proved to solve both of the  
3 above problems.

4 Figure 2, please, Bill.

5 (Slide 110.)

6 Figure 2 presents a comparison of the sift method  
7 and pile-and-draw method. Shown as the thickness of the final  
8 center plaque as a function of the final net weight of nickel  
9 powder laid down on a 2-square-inch sample, the result reveal  
10 a clear linear correlation between thickness and net weight  
11 of nickel powder laid down for the pile-and-draw method.

12 The sift method has no clear correlation.

13 Figure 3.

14 (Slide 111.)

15 Figure 3 presents plaques 100 square inches in  
16 size as a function of plaque weight for two groups of plaques  
17 produced on this machine. Of 1,010 29-mill plaques, 89 percent  
18 were within plus or minus 2 grams. Of 403 each 21-mill plaques,  
19 97 percent were within plus or minus 2 grams.

20 All of the plaques were 100 square inches in size.

21 This uniform weight characteristic is a direct  
22 result of the mechanics of the aforementioned pile-and-draw  
23 method.

24 The day-to-day production yield of plaques within  
25 plus or minus 1/1,000ths of a desired thickness is approximately

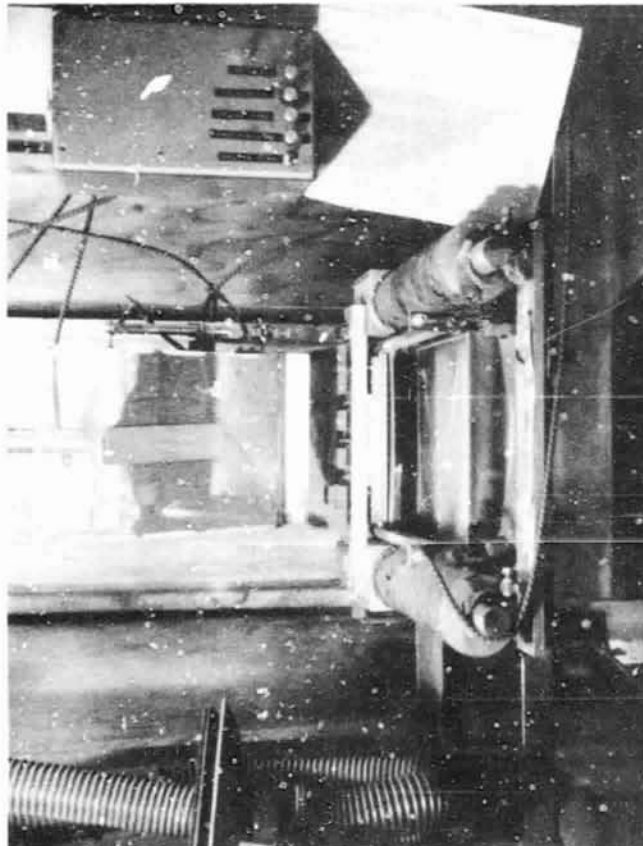


Figure 109

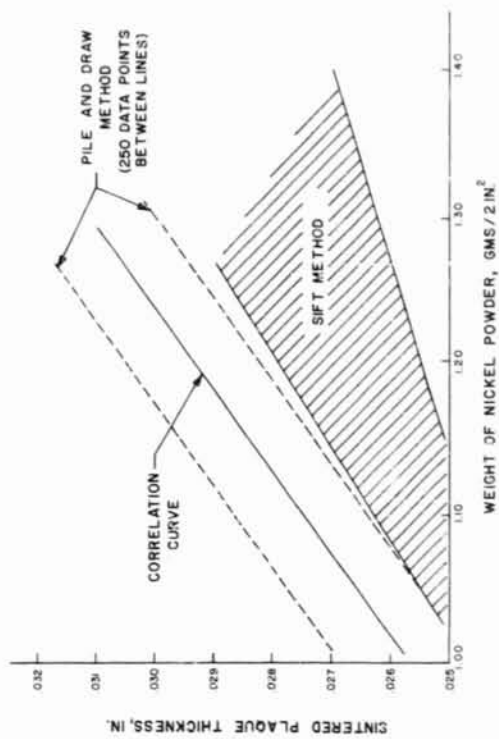


Figure 110 NUMBER OF PLAQUES AS A FUNCTION OF PLAQUE WEIGHT

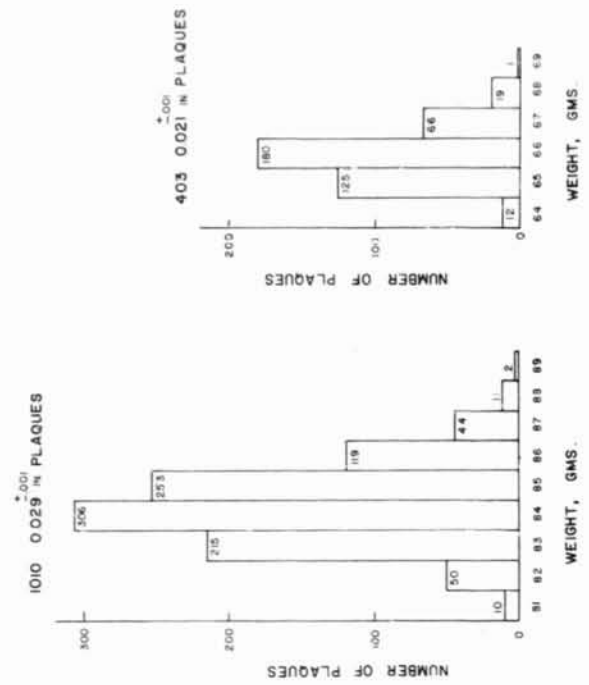


Figure 111 NUMBER OF PLAQUES AS A FUNCTION OF PLAQUE WEIGHT





Figure 109

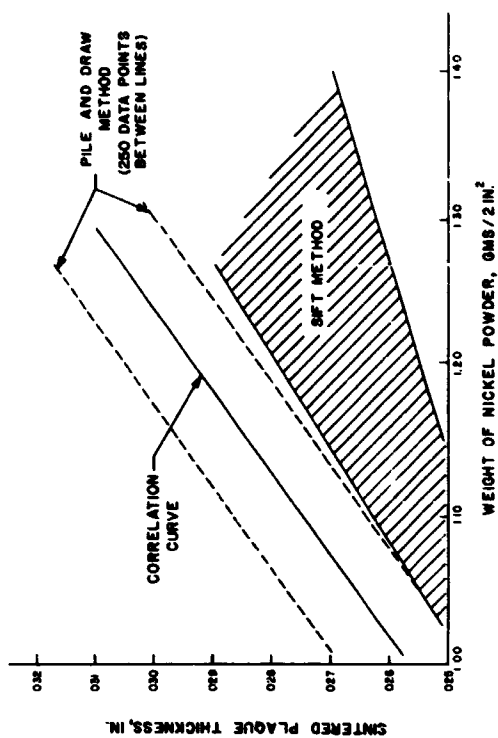


Figure 110 NUMBER OF PLAQUES AS A FUNCTION OF PLAQUE WEIGHT

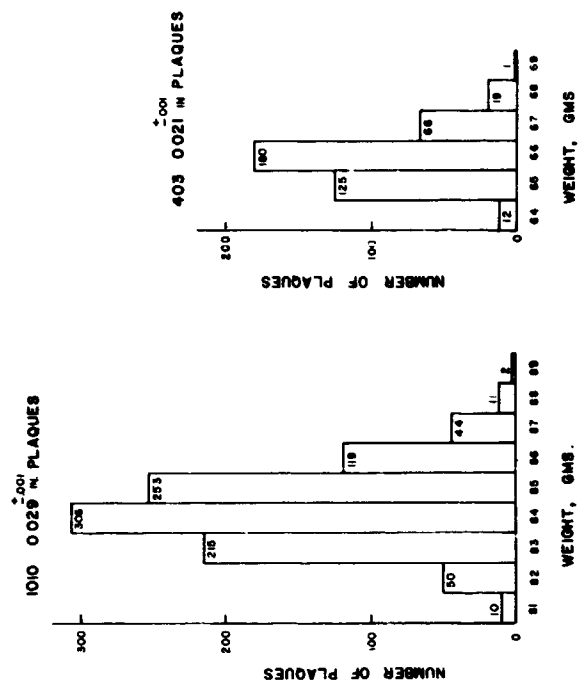


Figure 111 NUMBER OF PLAQUES AS A FUNCTION OF PLAQUE WEIGHT

eb8

1 90 percent. The porosity variation of 1,010 29-mill plaques  
2 previously mentioned was from 85.0 to 85.7 for the particular  
3 furnace conditions of the same process.

4 The strength of the plaques as determined by  
5 a standardized break test exceeds the normal requirements  
6 applied to similar products manufactured by the manual method.

7 From our data we reached these conclusions:

8 We concluded that a dry sintered nickel plaque  
9 can be produced by a machine system, and such a procedure  
10 yields desired characteristics of thickness, porosity, and  
11 weight per unit volume of area.

12 We might add that this process has been incorporated  
13 on the following programs:

14 Philco-Ford SMS program, Grumman 100 ampere hour  
15 program, NASA/Goddard 6 ampere hour program for separator  
16 study, and Boeing cells for the United States Air Force.

17 May we have the lights, please?

18 (Slide off.)

19 HALPERT: Are there any questions for Bob about  
20 this process?

21 John?

22 PARRY: Parry, Arthur D. Little.

23 Is there any tendency for the nickel powder to  
24 stick to the nickel platen?

25 HOWARD: Yes, sir, there is.

eb9

1 PARRY: Does this cause any real problem from the  
2 point of view of the production process?

3 HOWARD: The carrier platen -- nichel carrier  
4 platen is treated in such a way that this sticking is only  
5 around the edges of the platen. The plaque is then trimmed  
6 and this area is cut off.

7 PARRY: What is the treatment?

8 HOWARD: Zirconium oxide.

9 PARRY: Thank you.

10 HALPERT: Any other questions?

11 Okay, thank you-- Oh, sorry, one more back there.

12 BACHER: Are there any problems with porosity  
13 variations through the thickness of the plaque?

14 HOWARD: This was investigated with a weight  
15 determination of various areas over the plaque wherein areas  
16 were punched from the plaque and carefully analyzed for weight.  
17 We have found that the distribution of weight per unit area  
18 is very good in this dry sintered plaque.

19 BACHER: I don't mean from an area point of view  
20 but is one side of the plaque denser than the other side of  
21 the plaque?

22 HOWARD: Predominantly the substrate structure is  
23 on one side of the plaque. This would possibly limit surface  
24 area on that side, yes, sir. Is that the question?

25 BACHER: Yeah. Well, could this affect the surface

abf0

1 area that the opposing negative plates see -- you know, the  
2 opposing plates?

3 HOWARD: I think Earl Carr is just chomping at the  
4 bit to answer that question.

5 CARR: Earl Carr, Eagle Picher.

6 The reason that the screen wire is predominantly  
7 on one side is because in order to maintain something similar  
8 to previous qualified designs we intentionally put the screen  
9 wire off center on one side.

10 Now this procedure is so good that you can  
11 literally laminate the screen on one side of the plaque and  
12 then all you see is the screen and the plaque almost looks  
13 like it's separated -- like it's glued on, but that's a  
14 deviation from our process.

15 Now we're continuing on to investigate putting the  
16 screen in the precise center of the plaque. Again, this is a  
17 change to what has been used in the past so we have to proceed  
18 a little bit slowly.

19 But I have a question, and that is I understand  
20 your question in that the variation, say, over the thickness,  
21 whether it's a 20-mill plaque or a 30-mill plaque, what is it  
22 at one point and what is it at another.

23 We really don't know a good way to measure this  
24 and if anyone has any comments about this we would really  
25 appreciate any input as to how you would determine the uniformity

ebf1

1 of a cross-section like that.

2 HOWARD: Also in response to your question, we have  
3 analyzed fixed areas of an impregnated plaque and the net weight  
4 per fixed area is consistent over different areas of the plaque  
5 However, analyzing for which side and how deep the impregnation  
6 is -- I'm not sure I can answer your question, sir.

7 CARR: Also, regarding the effective area of the  
8 plaque, if you were to take the plates and when you orient  
9 them in the cell you will find that the screen side of the  
10 positive always opposes the screen side of the negative so that  
11 you always have the like side opposed to like side in a  
12 design.

13 I think you can work this out. It's a geometrical  
14 thing. The plates are all the same so when you turn the nega-  
15 tives against the positives they face the same type side.

16 HALPERT: You're telling your secrets.

17 Any other questions of Bob?

18 GROSS: Sid Gross, Boeing.

19 Have any measurements been taken on the strength of  
20 the plaque?

21 HOWARD: Yes, sir, there has been.

22 GROSS: Do you have any comments on how strength  
23 is changed as a result of modifying the processes?

24 HOWARD: I would say that the strength is more  
25 uniform over the given area of the plaque. As far as

eb12 1 mechanical strength, this is determined by the sintering  
2 elements of the process, not by the interaction of the long  
3 nickel fiber and any matter that would be just deposited on the  
4 substrate structure.

5 As far as the strength determination, we have the  
6 designer of this machine, I believe when he was with Tyco Labs.  
7 If you have any questions about the elements of the machine  
8 or the method of determination of strength, he would be glad  
9 to answer those, I'm sure.

10 VOICE: Thank you, Bob.

11 HOWARD: That's fine. I'll just shift a little of  
12 the responsibility around.

13 Yes? One more question?

14 RUBIN: Rubin, Tyco.

15 You mentioned some weight distribution over a  
16 hundred-square-inch plaque, 85 percent of them fell within two  
17 grams of a certain net distribution. What is the smallest  
18 area that you measured and what was the weight distribution on  
19 that smallest sample?

20 HOWARD: Weight distribution for a given area?

21 RUBIN: In your method you distribute a fairly  
22 consistent amount of powder over a large area, but have you  
23 measured the uniformity over smaller areas and if so, what  
24 was it?

25 HOWARD: Yes. The measurements mentioned were

eb13

1 over a smaller area. They were two-square-inch samples, to  
2 about one inch effectively, and these were cut from the same  
3 areas of different plaques and then compared with each other  
4 on plaques and with each other, plaque to plaque --

5 RUBIN: And this gave --

6 HOWARD: -- by thickness and weight.

7 RUBIN: And what percentage deviation did you find  
8 in those small areas from plaque to plaque?

9 HOWARD: I'm sorry, I don't think I have that data  
10 with me. I don't really know.

11 CARR: Earl Carr, Eagle Picher.

12 The reason we went into this work was as a result  
13 of the first two quarters of the NAS 521159 contract. We found  
14 variation within-- We were working to plus or minus 3/1,000ths  
15 which is reasonably typical and we found variation within  
16 this range and we identified it.

17 And we found some surprises during the first two  
18 quarters in that we took the data and we compared within a  
19 plaque nine samples, as Bob mention, two-square-inch samples  
20 taken in nine places from a plaque. Then we compared those  
21 nine samples to each other; we compared them to the next plaque  
22 in the sequence. Then we compared them to the next day at the  
23 same setting, and then we compared them to the next week; in  
24 other words, different types of reruns.

25 And the surprise was -- and this data is presented

NV-4

1 in the first two quarterly reports -- was that we found that  
2 the variation in the plaque is in any one single plaque.  
3 Something like 80 percent of all the variation we ever experienced  
4 you can find -- or we used to be able to find in a single  
5 plaque, so that it became very obvious that, if you have a  
6 plaque, a single plaque, that does not have any variation,  
7 we have immensely improved the process, and this, indeed, is  
8 what we did.

9           The means by which we did it were to lay a plaque  
10 which, when it is introduced into the furnace, is uniform  
11 because, if it is introduced into the furnace uniformly, it  
12 comes out uniformly. So this is why the emphasis was placed  
13 on the equipment to lay the plaque in a uniform manner.

14           To maybe add one more point on your question, in  
15 Figure 2, I presented the weight of nickel powder in grams per  
16 two-square-inch sample. This was on 250 data points cut from  
17 various plaques, possibly from -- I would assume maybe five  
18 samples per plaque or something along that line so there would  
19 be an analysis across approximately 500 square inches of  
20 plaque area.

21           It appears that there may be less than maybe one  
22 .2 of a gram variation among the samples pulled from 500 square  
23 inches of plaque material.

24           MAHATO: Mahato, from Globe Union.



eb15

1 Is that variation any way related to the particle  
2 size?

3 HOWARD: I'm sorry, I didn't understand the question.

4 MAHATO: Is that variation any way related to the  
5 particle size?

6 HOWARD: The particle size of the nickel powder?

7 MAHATO: That's right.

8 HOWARD: An analysis on the individual size of each  
9 nickel fiber laid down wasn't done but the bulk density of the  
10 powder prior to its laying on the substrate was analyzed.

11 Other than the bulk density measurement, I'm not sure I can  
12 answer your question about particle size of the nickel fiber.

13 However, there are two types of carbonyl nickel  
14 powder. Both were investigated and both can be satisfactorily  
15 laid in the dry process.

16 BELOVE: Belove of Marathon.

17 Was the bulk density of the various cells main-  
18 tained at a constant?

19 HOWARD: Yes, sir. The bulk density does come in  
20 in a varied range within one particular type of the carbonyl  
21 powder. I believe that your carbonyl type 287 might run from  
22 .8 grams per cubic centimeter to as high as 1 gram per cubic  
23 centimeter.

24 The dry process allows us to control bulk density.

25 We can select our bulk density to any desired final plaque

ed16 1 characteristics, both within the 255 and the 287 range.

2 BELOVE: You select the bulk density. That means  
3 you select barrels of material and reject other barrels?

4 HOWARD: We may use all barrels received but bulk  
5 density can affect porosity of your plaque on a unit area --  
6 weight or thickness unit area.

7 BELOVE: Thank you.

2 8 HOWARD: I would like to also point out this is one  
9 advantage, we feel, in the dry sinter process, is the ability  
10 to control bulk densities. It selects rather nicely to what-  
11 ever you want to get out the other end of your furnace.

12 FORD: Ford, NASA/Goddard.

13 Gerry, I would like to ask one question.

14 Bob, if I understand what you're saying you're  
15 saying that the variable is in the bulk density but by con-  
16 trolling some of the variables in the process, you can account  
17 for this in the final plaque?

18 HOWARD: Do you think you remember that well enough  
19 to repeat it?

20 (Laughter.)

21 FORD: Okay. I'm trying to clarify a point because  
22 I'm a little bit confused now.

23 You acknowledge there is a variable in the bulk  
24 density of nickel powder.

HOWARD: Yes, sir, there is.

eb17<sup>5</sup>

1 FORD: And the implication of what you said is  
2 that you allow this variable to come into the sintering  
3 process but you can counteract it somewhere in the process to  
4 give the same plaque characteristics in the end product.

5 HOWARD: Yes, you can, Floyd. It is possible that  
6 you can change your furnace setting to allow for deviations in  
7 bulk density, --

8 FORD: Okay.

9 HOWARD: -- but I'm not sure that is what you'd  
10 want to do. I think I'd rather know what bulk density I'm  
11 starting with and leave my furnace where I had it and take  
12 the sacrifices on the higher end of the bulk density and  
13 porosity.

14 PARRY: Parry, Arthur D. Little.

15 The work we did at Tyco, I think we found that if  
16 there were variations in bulk density with the starting powder,  
17 that if you did change either the sintering time or the sinter-  
18 ing temperature you could end up with a plaque with the  
19 characteristics that you wanted.

20 We would need some exploratory work to find out  
21 what you had to do to get the characteristics you wanted but  
22 in general, the porosity, the resistance, the mechanical  
23 strength of the plaque all move together so you could get the  
24 plaque with the characteristics you wanted just by changing the  
25 sintering conditions.

ebf8

1 HOWARD: I think Sonotone is going to have some-  
2 thing to say.

3 BELOVE: Belove of Marathon.

4 I didn't want to pursue this thing but now that it  
5 has been taken up, the powder variation -- the powder as we  
6 receive it varies practically from barrel to barrel, and  
7 probably within the barrel. The question here is-- There are  
8 several ways of maintaining uniformity of the finished plaque  
9 if the bulk density of the powder is found to vary.

10 The point we're trying to get here is: How do you  
11 obtain uniformity in the plaque, when the barrel -- the bulk  
12 density of the various barrels vary?

13 One method of course is to mix the powder and  
14 obtain a uniform or at least a practically uniform bulk  
15 density. Now the other method would be, as we just mentioned,  
16 by changing the parameters which would be a rather tricky  
17 job because it would mean experimenting on every barrel of  
18 powder, practically.

19 Now the question is: How do you do it in a practi-  
20 cal regime?

21 HOWARD: Day by day I control my bulk density. I  
22 don't blend to a bulk density.

23 BELOVE: You don't blend. You control the bulk  
24 density?

25 HOWARD: Yes, sir. I utilize bulk densities within

eb19

1 a range that I find acceptable.

2 BELOVE: That means that you reject barrels of  
3 powder back to the vendor?

4 HOWARD: I haven't lately.

5 (Laughter.)

6 BELOVE: Then Eagle Picher must have a contract with  
7 Inco that's special.

8 HOWARD: We have a working relationship with those  
9 people.

10 BELOVE: That's good to know. Now I've learned  
11 something. Thank you.

12 (Laughter.)

13 PARRY: Parry, Arthur D. Little.

14 Again my experience was that within the barrel,  
15 the bulk density was pretty uniform. We used to sample the  
16 barrel at various levels in the course of doing our work and  
17 for us, a 200 pound barrel would last maybe three or four  
18 months for the work we were doing, so this was over quite an  
19 extended time period.

20 BELOVE: I would out that for you that's all right.

21 PAPRY: This is true. I think you have a very  
22 valid point, that you have to experiment for each bulk density  
23 if you want to control the characteristics of the final plaque.

24 There are variations from barrel to barrel, but I  
25 would like to mention, too, that blending powders is not the

eb20<sup>11</sup>  
1 solution to the problem because these nickel carbonyl powders  
2 are relatively fragile and any blending process you go through  
3 actually changes the bulk density.

4 BELOVE: That depends on the quantity.

5 HOWARD: I might add that that's my experience,  
6 also. I have had no luck in blending nickel powders.

7 BELOVE: Again, it depends on the technique used.

8 HOWARD: I'm sure that's true.

9 CARR: Carr, Eagle Picher.

10 We made a little booboo a long time ago on doing  
11 the work, the same mistake that John just stumbled into. His  
12 powder lasted him three or four months.

13 When we set up the first regression models for  
14 the sintering process we did not consider, just as an over-  
15 sight, the bulk density. Lou Belove has made a statement  
16 that it's very tricky. It can be done a lot easier than most  
17 people think by regression analysis techniques to establish  
18 precisely the variations in furnace setting to compensate for  
19 a change in bulk density.

20 But these things are related to things like  
21 furnace atmosphere, dew point, like you wouldn't believe, so  
22 it is tricky but it is within the state of the art to pre-  
23 cisely know what settings will give you what porosity.

24 BACHER: Joel Bacher, RCA.

25 To change the subject slightly, among the plaque

eb21

1 parameters that you measured you mentioned porosity, weight  
2 and thickness. Do you have any means of determining degree  
3 of fusion or sintering or some strength?

4 HOWARD: Yes, sir, we do. It's a four-point band  
5 tester developed by -- or designed I guess originally by  
6 Tyco. And the designer, the original designer of that machine  
7 is with us today, and he'll be glad to comment on that if  
8 you'd like.

9 (Laughter.)

10 I think he is going to refuse to comment right now.

11 PARRY: Parry, Arthur D. Little.

12 I just want to make one comment to Earl Carr. I  
13 didn't --

14 HOWARD: This is the designer, by the way.

15 (Laughter.)

16 PARRY: The four-point band test works and it's  
17 fully written up for anyone who wants to look it up. I make  
18 no further comment on that.

19 I would like to say to Earl Carr that I didn't  
20 exactly stumble onto the problems of blending nickel powders;  
21 it was a specific study on our part, and is also written up  
22 in one of the early quarterly reports.

23 CARR: Earl Carr, Eagle Picher.

24 Sorry about that, John.

25 One thing else I wanted to say in answer to the

eb22

1 question from RCA is there is another technique for studying  
2 bonding and this is also reported in some of our early  
3 quarterlies and that is scanning electron microscope pictures --  
4 I can't say that word. But SEM photographs are fantastically  
5 revealing as to the properties of the plaque. You can literally  
6 look at it and see pore bonding or pore fusion or uneven  
7 fusion in the photograph.

8 And also I think these photographs are useful in  
9 determining such things as pore size distribution. It's a very  
10 powerful technique.

11 HALPERT: Thank you, Bob.

12 Earl, I'm glad you brought that up. I just happen  
13 to have a series of three conglomerate photographs on plaques,  
14 using the SEM and I'll just show you. One is an experimental  
15 model. One is a special version. Another one is a commercial  
16 product, and I will just show you the three to give you some  
17 idea of the variations you can get between plaque materials.

18 (Slide 112.)

19 This is the experimental model. You see it's a  
20 conglomerate so that it covers quite an area. I think it is,  
21 as I recall, at 250 X. Each slide, each photograph in the  
22 conglomerate was taken at 250 X. This is a 250 X pattern of  
23 a significant area of this plaque.

24 You can see a fair amount of porosity but quite  
25 a bit of nickel powder bonding.



eb23

1 (Slide 113.)

2 Here's a second version. This was made more for  
3 aerospace use. This particular dark area here is a problem  
4 with the SEM. That particular part did not come out. It is  
5 not a hole area, as you can see.

6 The other areas that are porous tend to show up  
7 rather well but that's kind of blurred. But here you see  
8 slightly greater porosity; still quite a bit of nickel.

9 Both of these have been made in the slurry process.

10 (Slide 114.)

11 And the third is one which was sent to us as a  
12 commercial item and it is from the dry process. You can see  
13 here greater porosity. It's hard to talk about uniformity  
14 when you talk about plaque materials and I don't want to say and  
15 I can't say which is better for what purpose.

16 I think we have found that if you sinter them long  
17 enough, as John Parry and others have been saying, at different  
18 temperatures you can get the overlapping even greater, but  
19 this particular one seems to have greater porosity than the  
20 others. But as Earl has mentioned, it is a valuable tool for  
21 looking at these various plaque materials.

22 However, the mechanical strength test does give  
23 you the degree of bonding that you have between the nickel  
24 particles.

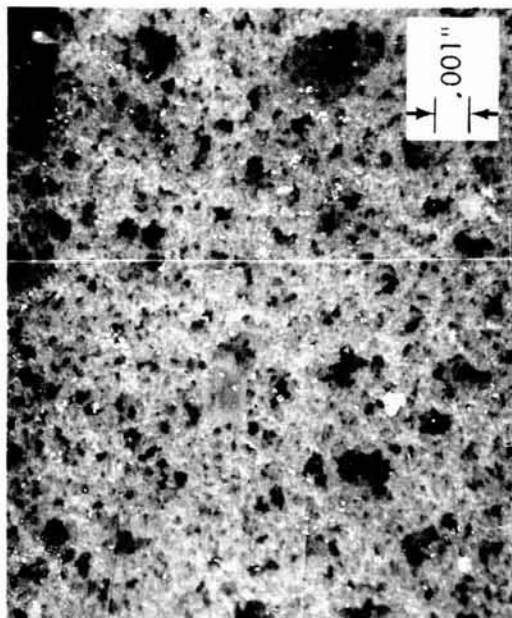


Figure 112

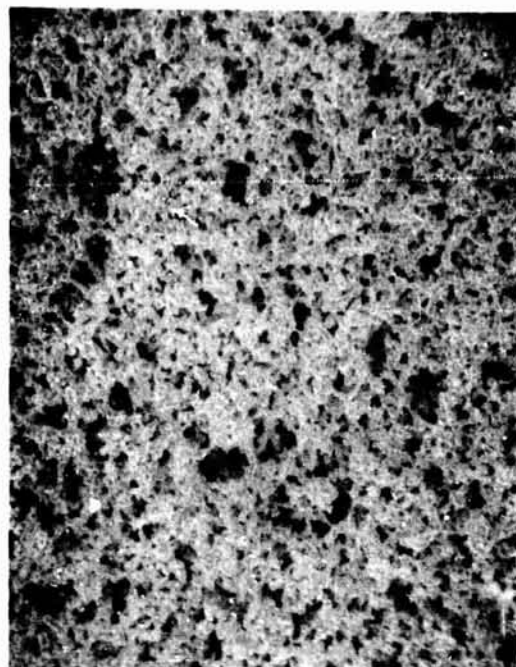


Figure 113

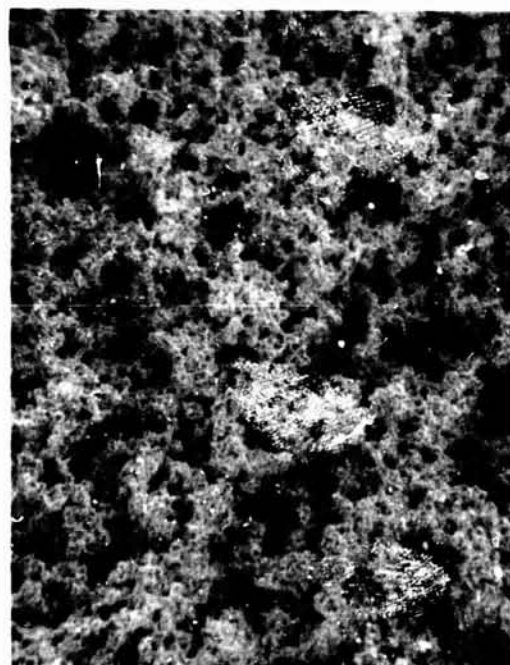


Figure 114

WT. BEFORE IMPREGNATION (grams)	WT. AFTER	NET GAIN
9.2222	16.9090	7.6868
9.3976	17.4829	8.0853
9.3192	17.2953	7.9761
9.2522	16.3804	7.1282

Total Wt. Gain = 30.5824

(Corresponds to 8.93 A-h the O. Cap.) 0.030 in. 83% porosity  
plaque 3" x 3", coined 0.010"

Figure 115

eb24-

1 Tom Hennigan -- I'm sorry, Tom -- wanted to ask a question.

2 HENNIGAN: I'd like to ask this of Ed Rubin, who  
3 used to work for TI, and this question came up before, can we  
4 get the grid in the center of the plaque?

5 If I remember right, TI's grid was on the side. It  
6 had to be on the side because of the process. This appeared  
7 to be satisfactory.

8 Do you have any comment? Were there any problems  
9 with this plaque with the grid on the side?

10 RUBIN: Rubin, Tyco.

11 In most of the plates that TI had worked with, they  
12 were under 30/1,000ths thick, and restricting ourselves to that  
13 thickness, we did not run into any difficulty in terms of  
14 swelling of materials or warpage of the plates.

15 I believe once you get into thicker plates than  
16 that you will run into some difficulty, especially with warpage.

17 Of course when it comes to making cylindrical cells  
18 it is desirable to have a screen on one side.

19 HALPERT: Are there any other comments or questions  
20 regarding plaque materials?

21 (No response.)

22 HALPERT: Okay, if not, then we'll go over to the  
23 next step, that is in the plate materials themselves, impregna-  
24 tion, and David Pickett of the Aero Propulsion Lab., Wright-  
25 Patterson, wants to talk about nickel hydroxide electrodes

eb25  
xzxzx

1 fabricated from the Bell high temperature electrochemical  
2 impregnation process.

3 PICKETT: One of the first presentations at the  
4 battery workshop last year was given by Dick Beauchamp of  
5 Bell Labs on a new high temperature electrochemical impregna-  
6 tion process for a nickel-cadmium electrode. In this process  
7 a sintered nickel plaque is impregnated by making the plaque  
8 cathodic and immersing the plaque into a boiling solution of  
9 nickel or cadmium nitrate between two inert annodes and pass-  
10 ing a current of about a half amp per square inch of electrode  
11 surface through the plaque.

12 The pH of the solution was initially set at about  
13 4 and was controlled by an initial addition of sodium nitrate  
14 to the impregnating solution.

15 Electrodes made by this process showed a substantial  
16 increase in capacity over electrodes made by conventional  
17 vacuum impregnation techniques. In fact, performance of the  
18 electrodes was quite impressive in general.

19 Since this time we at the Air Force Propulsion Lab,  
20 especially Dr. Lander and I, have been investigating the  
21 process in one of our in-house efforts. I would like to  
22 present to you today some of the data that we have obtained  
23 from this. It is by no means conclusive but I think some of  
24 it is rather interesting.

eb26

1 Bell Labs has introduced papers, in particular at the Electro-  
2 chemical Society in Cleveland, where they use various additives  
3 in the process for the positive plate such as cobalt.

4 In the original impregnation solution, two molar  
5 nickel or cadmium nitrate and .2 molar sodium nitrite solution  
6 was used and impregnation was carried out on inch and a half  
7 by inch and a half plaques. We deviated-- Well, excuse me.  
8 These were connected in series in beakers so if you wanted to  
9 impregnate several plaques at a time you merely connected the  
10 beakers in series.

11 We deviated from this original procedure by carry-  
12 ing out our impregnations in a large Pyrex vessel, connecting  
13 all of the plaques to be negative of a power supply and all  
14 of the annodes to be positive so essentially we have the whole  
15 thing in parallel -- plaques connected in parallel in the large  
16 bath.

17 Our solution contains 1.8 molar nickel nitrate,  
18 .2 molars cobalt nitrate, and .2 molars sodium nitrite. The  
19 current we used varied from .3 of an amp per square inch to  
20 .5 of an amp per square inch, and in most of our successful  
21 runs we have impregnated plaques as high as -- using currents  
22 as high as one amp per square inch.

23 I would like to show you some of the gains that  
24 we have obtained on these.

(Slide 115.)

eb2-7  
1           These are 30 mill plaques, dry sinter variety, made  
2 by Eagle Picher, and you can see that the total weight gain  
3 varies between 7 and 8 grams. This corresponds to roughly 8.9  
4 ampere hours theoretical capacity, on the theoretical.

5           We formed plates of this type by combining in parti-  
6 cular these four plates with five negatives taken from a  
7 standard 22 ampere hour aircraft cell that were cut to 3 by 3  
8 dimensions. All nine plates were made up in a formation cell  
9 and the cell was given an initial formation charge at C over  
10 10 of the theoretical capacity with about 20 percent overcharge.

11           Discharge at the one hour rate to .9 of a volt  
12 showed 6.55 ampere hours capacity. The cell was then charged  
13 at the one hour rate and discharged at the one hour rate to  
14 .9 of a volt for 27 more cycles and eventually showed a capacity  
15 of 10.4 ampere hours.

16           Positive plates from this cell were removed, placed  
17 with four other positives formed by the same process and the  
18 resulting cell placed on cycle life at 120 degrees Fahrenheit  
19 with nine negative plates, again the plates being taken from  
20 conventional ni-cad batteries.

21           The cell was sealed with a 26 psi release and  
22 initially charged at 20 amps for one hour. The first discharge  
23 showed 17.1 ampere hours to a cutoff voltage of .9 volt.  
24 Cycling was then carried out by charging at 33 percent over-  
25 charge at the one hour rate based on capacity from the previous

1 discharge to .9 volt.

2 A typical example of some of the capacities obtained:

3 At cycle No. 109, 19.7 ampere hours were put in  
4 and 13.8 ampere hours were obtained on discharge to .9 of a  
5 volt.

6 After 152 cycles, the cell shorted. We have made  
7 other cells by this method and have run them at these high  
8 temperatures. Eventually in some of these electrolyte was  
9 dried out but we have obtained much higher cycle life than these.  
10 But this is just a typical example of some of the work that  
11 we have done.

12 I think the most impressive thing about it is the  
13 charging efficiencies for this high temperature. We do not get  
14 near as good charging efficiencies out of conventional air-  
15 craft batteries at this temperature. In fact, they are well  
16 below 50 percent.

17 We are still doing investigations on cells of this  
18 type and are making negative electrodes by the Bell process.  
19 We hope to have some data on complete cells in the near future.

20 That is the essence of my presentation. Are there  
21 any questions?

22 LACKNER: Lackner, Canadian Defense Research.

23 You mentioned you got good performance at high  
24 temperature. What temperature and for what number of cycles  
25 were you operating out of?

eb29<sup>th</sup>

1 PICKETT: All these cycles were carried out at 120  
2 degrees Fahrenheit.

3 LACKNER: Charged and discharged at 120 degrees?

4 PICKETT: At 120 degrees.

5 LACKNER: For how long?

6 PICKETT: One hour. We measured the capacity to  
7 .9 of a volt on the previous discharge and then put in the  
8 same capacity with 33 percent overcharge at the one hour rate.

9 LACKNER: For the 150 cycles before it shorted out?

10 PICKETT: Right.

11 LACKNER: What kind of separator did you have in  
12 there?

13 PICKETT: We had a layer of cellophane and a layer  
14 of nylon.

15 LACKNER: Good enough.

16 PICKETT: Gerry asked if these were flooded. Yes,  
17 these are flooded cells.

18 MAURER: Maurer, Bell Laboratories.

19 Did this short occur because the electrolyte had  
20 dried out?

21 PICKETT: Apparently so.

22 PARRY: Parry, Arthur D. Little.

23 Dave, do you have a figure for the specific capacity  
24 of the positive plates as operated in the cell, the ampere hours  
25 per cubic inch?



eb30

1 PICKETT: Well, based on the figures that I've given  
2 you and after 28 or 29 formation cycles, the capacity was  
3 roughly 10 ampere hours per cubic inch of void volume available  
4 in the plaque.

5 PARRY: Are all your capacities on the basis of  
6 void volume or volume of the plate?

7 PICKETT: We usually calculate them on the basis of  
8 void volume in the plaque but these can easily be converted.

9 FONT: Font, of SAFT.

10 What can you say about the aspect of the positive  
11 plates compared to plates which were impregnated in vacuum  
12 condition with standard plates, if you will, the aspect of the  
13 positive in such process after such cycling?

14 PICKETT: Well, we --

15 FONT: Did they --

16 PICKETT: In terms of what? Cycle life or capacity?

17 FONT: Yes, cycle life and the flake of the plates,  
18 for example.

19 PICKETT: Well, it's really hard to compare cycle  
20 life here with other cells that we made because this is one of  
21 the first experimental cells we've made and tried, and it would  
22 be I don't think justified to really compare it with any other  
23 cells, but of course the other cells do get much greater cycle  
24 life.

25 As far as capacity goes, I would venture a guess --

eb31

1 I don't know exactly, but I assume that conventional state-of-  
2 the-art aircraft battery electrodes have about four + five  
3 ampere hours per cubic inch of electrode and that these would  
4 be roughly between eight and nine.

5 FONT: Thank you.

6 BELOVE: Belove of Marathon.

7 Dave, I heard you say you formed for 29 cycles.  
8 This formation occurred before you began testing the battery?

9 PICKETT: That's correct, yes.

10 BELOVE: What does the formation cycle consist of?

11 PICKETT: Our first cycle was a C over 10 charge  
12 based on the theoretical capacity of active material we had in  
13 the cell. This was then discharged at the one hour rate to .9  
14 of a volt.

15 The subsequent charges were made at the one hour  
16 rate and discharges made to approximately the one hour rate to  
17 .9 of a volt.

18 BELOVE: In all cycles thereafter, they were  
19 charged at the one hour rate?

20 PICKETT: That's correct.

21 MAURER: I would like to comment that at Cleveland,  
22 Beauchamps presented a paper on additive effects to improve  
23 cycle life on the electrodes made by these processes and one  
24 can get cycle lives of several thousand cycles, deep discharged  
25 when cobalt is present in the electrode.

eb32

1 FONT: Font, SAFT.

2 Have you performed the chemical analysis on either  
3 content -- hydroxide nickel content on your electrodes?

4 PICKETT: No, we have made no analysis as yet. We  
5 are in the process of doing that now and the results just  
6 weren't available for this presentation.

7 FONT: Thank you.

8 SULKES: U. S. Army Electronics Command.

9 After you finished the cycling did you take any  
10 physical data? In other words, particularly on these high  
11 utilizations did the plaques swell or have cracks or that type  
12 of thing?

13 PICKETT: There was some swelling, yes, but there  
14 were no cracks in the plaque.

15 SULKES: Would you have an idea of what, say, the  
16 actual ampere hours per cubic inch ended up after the swelling?  
17 In other words, I've had plaques from various manufacturers  
18 that, say, started out at 9 but by the time they finished  
19 swelling and growing, they were back at 6-1/2 and 7 where  
20 everybody else's plaque is.

21 PICKETT: Well, no, we didn't measure the thick-  
22 ness of the plaques after the 150 cycles, no. After the 29  
23 cycles we did. We showed no substantial increase after the  
24 29 formation cycles.

25 SULKES: Well, usually it would occur right away

eb33-

1 so you may have something.

2 BACHER: Bacher, RCA.

3 Getting back to the original plate processing, what  
4 were the inert electrodes?

5 PICKETT: In the Bell process they use platinum  
6 electrodes or platinized titanium. We do not use electrodes  
7 of that type and I don't guess you would call our electrodes  
8 inert. We use 200 nickel and there's substantial corrosion to  
9 the electrode.

10 HALPERT: Any further questions?

11 (No response.)

12 HALPERT: Thank you very much.

13 for the next subject, I have a short presentation on  
14 some negative plate materials that we've looked at from four  
15 ampere hours cells on test at Crane. One set of cells had a  
16 teflonated negative and another was non-teflonated, and Floyd  
17 I think has some background data that I think it might be  
18 helpful to present before I show the photographs that I have.

19 FORD: In the early stages of the SAS-A program,  
20 APL, which is the prime contractor on this program to Goddard,  
21 procured cells from two different manufacturers. For identi-  
22 fication purposes, if you are interested in following these  
23 tests further at Crane where the cells are still on test,  
24 Pack 1-C, "C" as in "Charlie," the manufacturer is General  
25 Electric; Pack 18-D, Gulton Industries, and one correction to

MV-5

1 what Gerry said, these are 6 ampere hour rated capacity cells.

2           These cells were put on a life cycle program for  
3 the SAS-D using a charge control system simulating that of the  
4 spacecraft. They were run in a 90-minute orbit. The depth of  
5 discharge was approximately 21 percent and they were run at 25  
6 degrees C. The charge current is limited at 3-1/2 amps until  
7 the voltage limit is reached, at which time it goes into a  
8 taper.

9           The voltage limit is 11.4 volts at 26 degrees C.  
10 This is a temperature compensating voltage. They were placed  
11 on life cycling on July the 22nd, 1970 and exactly one year  
12 later, one cell in each pack was removed for analysis. These  
13 are the cells Gerry will now discuss.

14           HALPERT: I don't have any chemical results at this  
15 point. These are really photomicrographs of samples of the  
16 materials, of the plate materials in the cell.

17           The first ones are from both cells. I'll try to  
18 get both of them in here at the same time.

19           (Slides 116, 117, 113.)

20           The left one seems to be a little out of focus.

21           All I wanted to show here: All are positives.

22 There was little or no difference. The bright parts that you  
23 see are the nickel plaque. These are cross-sectioned and  
24 mounted in sample holders. What you can see -- The grey area,  
25 the active material has come to the surface of the plate and

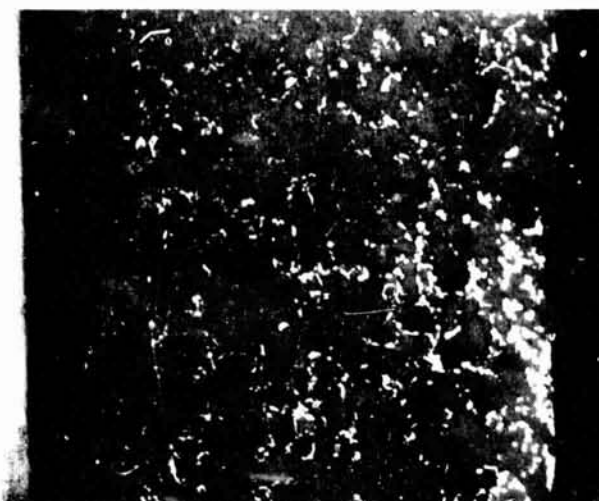


Figure 116

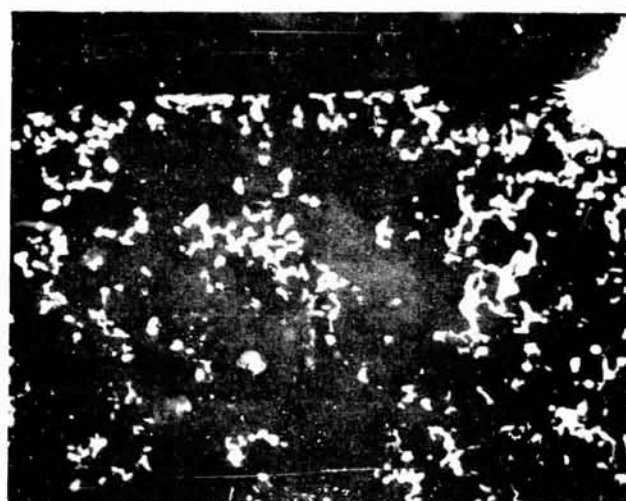


Figure 117

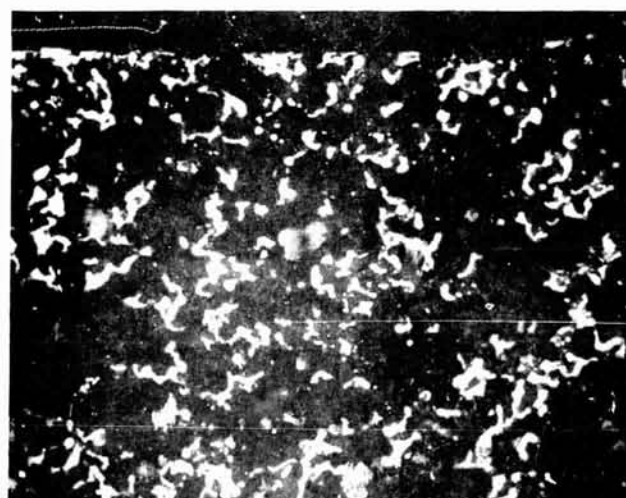


Figure 118

MV-6

1 it actually is not as clear in this particular photograph but it's  
2 the same. The active material has come to the surface of the plate

3 I have put in one of an unused material and I think  
4 here hopefully you can see that the active material is completely  
5 covering rather uniformly within the entire surface -- within the  
6 entire area of the plate rather than just accumulating at the "  
7 surface.

8 So I would say as far as deposit is concerned, in both  
9 cases we have -- Neither one was, of course, teflonated but in  
10 both cases the major change was the actual material near the  
11 surface.

12 (Slide 119.)

13 At the negative we tended to find in the non-teflonated  
14 large, fairly large crystals of negative material coming from the  
15 surface of the plate. I've got this first as a negative now at  
16 the surface, and that's a fairly large crystal. These photo-  
17 graphs are all 250 X (corrected from the 500 X cited by speaker.)

18 The very bright spots in this area are the nickel  
19 material for plaque. The gray area is the cadmium hydroxide,  
20 and then the shiny dots within the cadmium hydroxide is the  
21 cadmium active material.

22 You can see on this piece now that seems to be away  
23 from the surface of the plate that we have not only the cadmium  
24 hydroxide growing in a crystalline shape away from the plate  
25 but some cadmium metal also embedded in it.

NV-27

1 We found that at times removing a cell or removing  
2 a plate in the charge condition and wiping the surface of the  
3 plate, quite a bit of powder came off. We analyzed the powder  
4 and it was mainly cadmium metal and we did the same thing for  
5 discharge plates. The power was still very loose. We wiped  
6 it and took that material for sampling and almost all that  
7 material was cadmium hydroxide, which means that even though  
8 this powder is coming away from the surface of the plate, it  
9 is very loosely attached, it is still -- quite a bit of it  
10 is still electronically connected to the plate so it can be  
11 oxidized and reduced.

12 (Slide 120.)

13 You can see these two are the same or similar. What  
14 we've done is taken the sample and dipped it into the famous  
15 mussprat solution, affectionately referred to by our metallurgi-  
16 cal people as muskrat juice, and tried to remove some of the  
17 cadmium hydroxide, hopefully leaving some of the cadmium behind.

18 But, as you can see -- This was only a very short  
19 dip, as a matter of fact, but you can see that some of the  
20 cadmium has been removed, as well as the cadmium hydroxide in  
21 this dip process.

22 (Slide 121.)

23 This further photograph is one in which we used  
24 polarized light and here now you can see back in the area





Figure 119

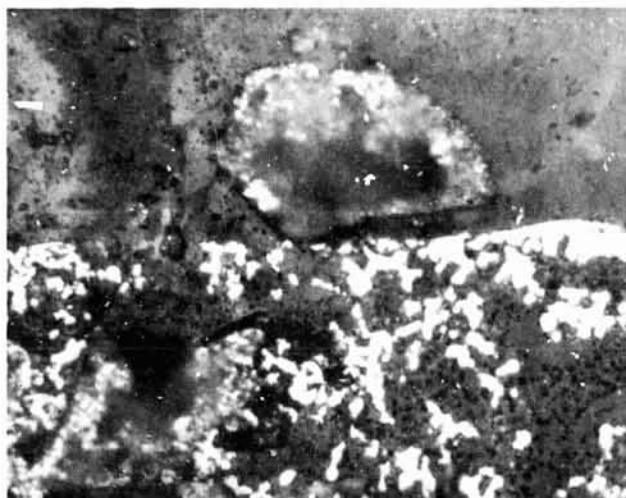


Figure 120



Figure 121

MV-3

1 here what looks like a small crystalline area. It comes out  
2 very bright and you get a kind of a 3-D effect in the crystal.

3 Up in here, you can see actual crystal into the  
4 mounting material away from the plate.

5 I have a couple more of these I can show, but, what  
6 it does do is bring out the crystals with greater detail.

7 (Slide 122.)

8 Okay. Now, looking at the separator material just  
9 adjacent to a sample of the plate, we found this -- As a cross  
10 sectional area we found this type of a structure in which you  
11 can actually see some of the cadmium and cadmium hydroxide  
12 particles.

13 These lighter gray spots are the nylon separator  
14 coming through but you can see both cadmium metal and cadmium  
15 hydroxide in those particles.

16 (Slide 123.)

17 This is another sample that was similar, actually  
18 dipped for a few moments in the musspratt solution.

19 (Slide 124.)

20 A further sample in which we dipped it in mussprat  
21 solution and you can see now these holes where some of the  
22 active material was that has now been removed, but if you look

23

24

ed38 1 at a polarized light photograph on the same sample, you can  
2 actually see the 3D effect.

3 (Slide 125.)

4 You can see the 3D effect in which you see the  
5 crystals right down into the separator material. Again, this  
6 darker area here is the nylon. These lines here are due to  
7 the nylon separator. What comes out-- Everything else is  
8 dark except the crystal material.

9 This is as I say in the non-teflonated one. We had  
10 similar results with the teflonated ones.

11 (Slide 126.)

12 This is a cross-section of the teflonated  
13 electrode in which again we see the nylon separator here and  
14 some of the crystalline material at the top. Now one of the  
15 things is, no matter which plate we used, we were able to find  
16 areas where there were large amounts of cadmium at the surface  
17 of the plate.

(Slide 127.)

18 In this particular case we found some further cadmium  
19 hydroxide by doing a polarized light.

20 Let's see if I've got the right direction. Yes, this  
21 is the same photograph in polarized light. You can see the  
22 crystalline material coming through very nicely, and we still  
23 have some crystalline material down in here although it seems  
24 to be lesser.

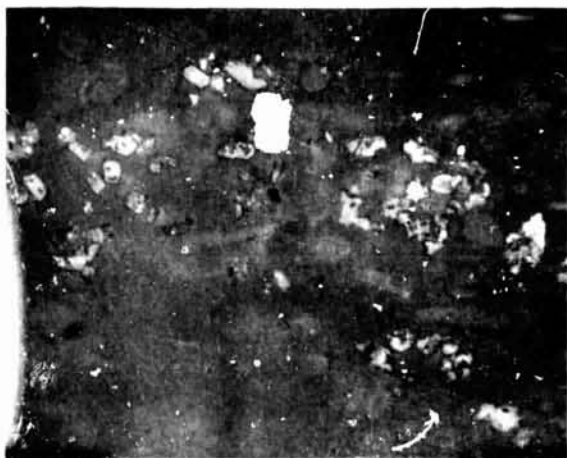


Figure 122

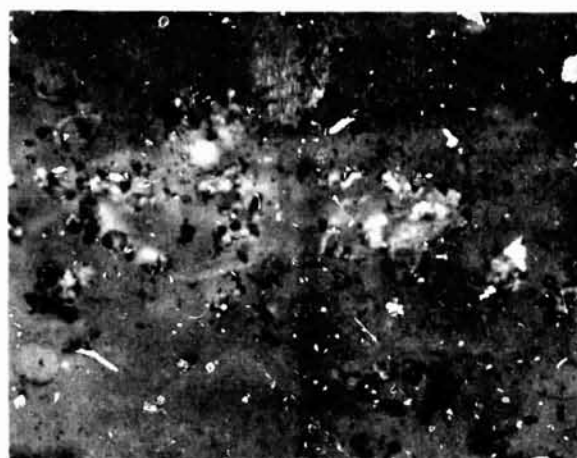


Figure 123



Figure 124

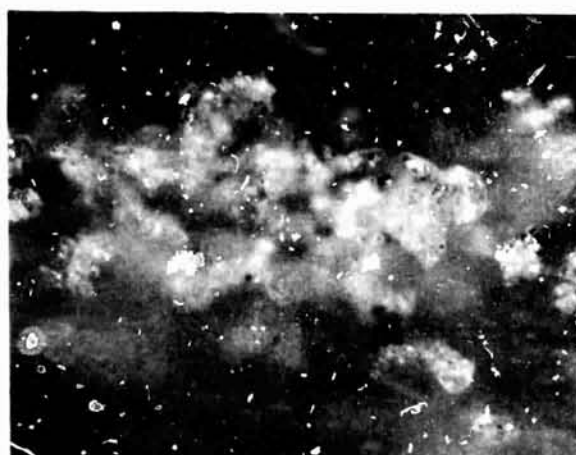


Figure 125

eb39

1 cadmium or small amounts of cadmium on the plate, on both types  
2 of plates, regardless of what it is teflonated or not. The  
3 major difference that we did find is the following:

4 Using the scanning electron microscope, we looked  
5 at only the surface properties of some of these materials and  
6 here we have a couple of photographs --

7 (Slide 128.)

8 -- of typical areas within the non-teflonated structure which  
9 seem to be larger and fairly irregular types of structures.

10 We can see here one of the nylon strands being  
11 embedded in that particular plate, but as I say, the major  
12 difference here was in this teflonated one-- That's the next  
13 one I can show you.

14 (Slide 129.)

15 You see a more regular shaped, smaller particles.

16 We don't know what the reason is yet. We do know that the  
17 separator pulls very easily from that negative electrode while  
18 in the case of the non-teflonated one we find that it takes  
19 quite a -- it does not come off easily. It can be washed off,  
20 of course, but it's more difficult. It seems to come off the  
21 negative.

22 In both cases we have some cadmium in the separator.

23 In the non-teflonated one we have a little bit more cadmium.

24 We found negative material on both positive plates in both  
25 cells so that the cadmium is getting through.

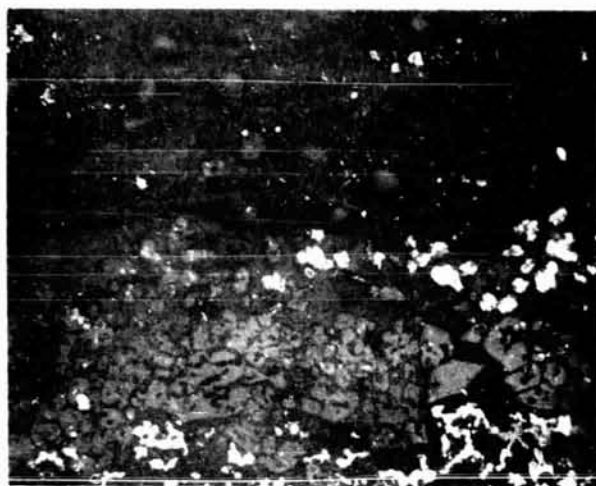


Figure 126



Figure 128



Figure 127



Figure 129

HV-9

1 It does appear in some of the samples we looked  
2 at to be lesser on the teflonated one than the non.

3 The main points at this time are two things: First,  
4 the separator came off very easily and, secondly, that this  
5 type of structure, another example of which is here --

6 (Slide 130.)

7 -- was fairly regular on the teflonated one while the larger,  
8 more irregular type crystal structure was available on the non-  
9 teflonated one.

10 Okay, that's it.

11 Are there any questions?

12 LACKNER: Lackner, Canadian Defense Research.

13 What do you mean by "teflonated" negatives?

14 HALPERT: I am told there are cells built which  
15 have teflonated negatives -- negatives which have teflon  
16 coatings on the plate.

17 Now, we did do an electron microprobe analysis to  
18 see if we could find the chlorine or the fluorine from the  
19 teflon on the surface of the plate and -- Since it was such  
20 a thin film and, since the plates may have been used for some  
21 time, it was very difficult to find where that teflon was and  
22 how uniformly it was put on, whether it had migrated into  
23 the center of the plate, or what.

24 So, thus far, we still don't know where the teflon is



Figure 130



eb41

1 or what actual purpose it's serving, other than to have that  
2 different crystal structure and remove the separator from the  
3 plate.

4 But what the process is in putting the teflon, I  
5 myself do not know. Maybe there is somebody here who might  
6 want to comment on that.

7 LACKNER: Who put it on?

8 HALPERT: The manufacturer put it on. These were  
9 cells that were prepared and have been under test for what? --  
10 a year, Floyd said.

11 FORD: Yes.

12 LACKNER: Who was the manufacturer?

13 HALPERT: General Electric was the manufacturer.

14 FORD: General Electric.

15 BELOVE: Were they sintered plates?

16 HALPERT: Yes, sintered plates made in the normal  
17 GE production as were the plates from the Gulton cells that  
18 were compared with it.

19 HAINES: Defense Research.

20 Would you like to comment on the effect that tef-  
21 lonating has on the absorption of electrolyte and on what effect  
22 it has on the oxygen recombination reaction?

23 HALPERT: I can't. Unless Floyd has some data on  
24 pressure, I don't have anything on that yet.

25 Do you have anything?

e342 1 FORD: I have no data prepared for this meeting. I  
2 will say this, that all these cells, both groups of cells,  
3 went through the acceptance test in which we have certain  
4 maximum pressure requirements and certain overcharge test,  
5 and all these cells met these requirements and they were  
6 acceptable as flight hardware.

7 BACHER: Bacher, RCA.

8 A little more, Floyd, for you. Has the teflon--  
9 Does it affect the electrical performance or cycling performance?

10 FORD: We haven't seen any degradation in capacity  
11 of these cells that we can attribute to the fact that there's  
12 teflonated negatives in the cells.

13 RAMPOL: Guy Rampel, General Electric.

14 I'll volunteer the information to that gentleman  
15 over there from the Canadian Defense that teflonated negatives  
16 in cells do require a little less electrolyte and pressures  
17 are lower.

18 HALPERT: Any further questions?

19 (No response.)

20 HALPERT: Okay, I think at this point we'd be due  
21 for a coffee break and we'll meet back about eleven o'clock.

22 (Recess.)  
23  
24  
25

B/5<sup>4</sup> ebl 1

2 seats?

3 I did want to make one comment with regard to my  
4 last presentation and that is I did say that the musspratt  
5 solution removes the cadmium and cadmium hydroxide. It doesn't  
6 attack the cadmium; it only attacks the cadmium hydroxide but  
7 since the cadmium hydroxide may be surrounding small particles  
8 of cadmium, the cadmium is removed at the same time.

9 Okay, as we continue on through this manufacturing  
10 materials session, our next speaker is Ed McHenry from Bell  
11 Labs who is going to talk about the scale-up studies of the  
12 Bell High Temperature Impregnation Process.

13 Ed McHenry.

xzxzx 14 MC HENRY: This is a report on some of the process  
15 studies we have done as a background toward a continuous  
16 impregnation process. We were just trying to gather some data  
17 and find out some of the problems we're going to have if and  
18 when we do attempt to scale this up and make a continuous  
19 process out of it.

20 The first slide, please?

21 (Slide 131.)

22 This is just a schematic of the process. Depending  
23 on whether you're making a nickel or cadmium plaque you use  
24 cadmium nitrate solution or nickel nitrate solution. The  
25 center plaque is cadmium or nickel hydroxide deposited by

eb2<sup>1</sup>

1 cathodic precipitation, washed, dried, and then put into  
2 formation.

3 Next slide, please.

4 (Slide 132.)

5 These are the compositions of the impregnating  
6 solutions that we used during this work. The nickel solution  
7 was a two molar nickel nitrate, tenth molar and cobalt nitrate,  
8 .3 molar and sodium nitrite, and the pH of 4.

9 Now this is the starting point when you first  
10 start your impregnation. What happens, as you impregnate your  
11 nickel is deposited as nickel hydroxide so your nickel con-  
12 centration is going down. The nitrate-- Presumably the  
13 reaction is not well understood but the nitrate would be re-  
14 duced at the cathode and then at the anode, some of your  
15 nitrite is oxidized to nitrate. Possibly some of the nitrite  
16 is also reduced at the cathode.

17 The cobalt co-precipitates with the nickel and  
18 pretty generally precipitates in the same ratio as it exists  
19 in the solution. In other words, if you have 5 percent cobalt  
20 in the solution-- In other words, 5 percent of the nickel,  
21 the weight of nickel, is cobalt, so that they will then pre-  
22 cipitate in the same ratio. All that you are doing is pre-  
23 cipitating by raising the pH, and all the hydroxide in that  
24 area, all the ions which -- where the hydroxide is insoluble  
25 will simply precipitate so it doesn't really matter what the

eb3-

1 electrode potentials are for deposition of these metals. You  
2 aren't depositing; you're simply raising the pH and so they  
3 just precipitate.

4 In the cadmium solution of course the nitrate is  
5 being reduced at the cathode and the nitrite is being oxidized  
6 at the anode.

7 In general, the nitrate concentration remains  
8 reasonably constant, the nitrite is going down, and the nickel  
9 and the cadmium are going down, and the cobalt is going down.  
10 The pH has a tendency to rise a little but as you run out of  
11 nitrite then the pH begins to go down.

12 Next slide, please.

13 (Slide 133.)

14 This is the impregnation jig that we used in this  
15 work. We used a 4 by 4 inch plaque as you can see in here,  
16 the black part. We used these platinized titanium counter-  
17 electrodes. This was for economic reasons. Oh, it's a factor  
18 of 10 cheaper to buy this type of material.

19 The jig that holds the electrode is made such that  
20 the working electrode is masked so that you have the current  
21 flow, the potential gradient, is perpendicular to the surface  
22 of the plaque so you get a uniform current density over the  
23 whole surface.

24 The next slide, please.

25 (Slide 134.)

# SCHEMATIC OF ELECTROCHEMICAL IMPREGNATION PROCESS

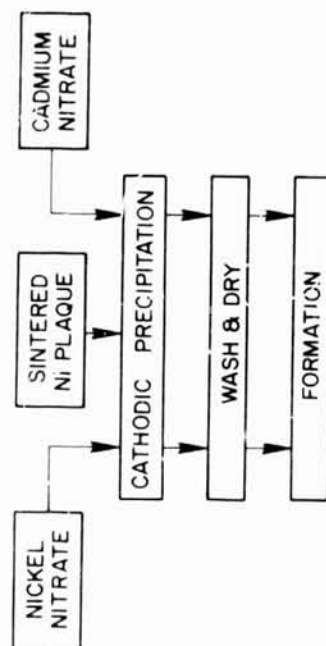


Fig. 131

## IMPREGNATING

### SOLUTION COMPOSITION

<u>Ni</u>		<u>Cd</u>	
2.0M	$\text{Ni}(\text{NO}_3)_2$	2.0M	$\text{Cd}(\text{NO}_3)_2$
0.1M	$\text{Co}(\text{NO}_3)_2$	0.5M	$\text{NaNO}_2$
0.3M	$\text{NaNO}_2$	pH = 4	
pH = 4			

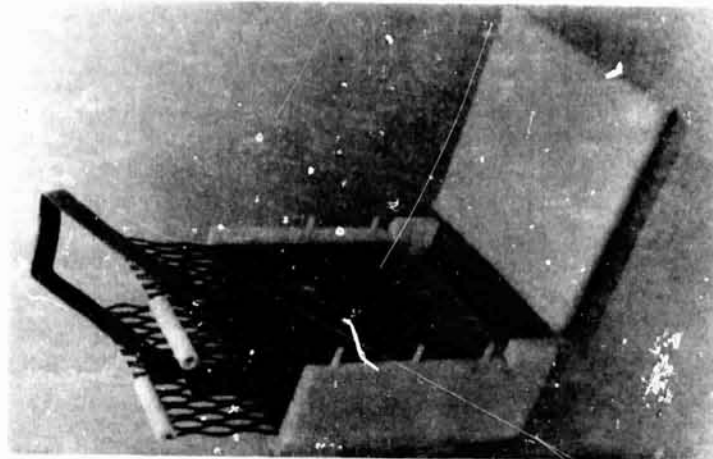


Fig. 133

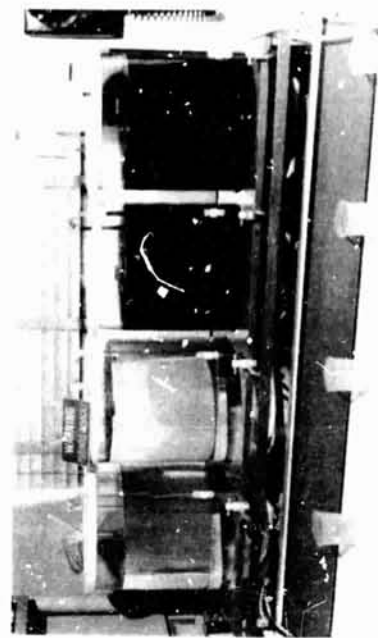


Fig. 134

eb4

1 Here we show the impregnating solutions that we  
2 use. We had two of each so that we could use the first one  
3 and then while we were analyzing and adjusting this solution,  
4 we used the other one, and we'd run on alternate days.

5 We ran-- Let's see--

6 Next slide, please.

7 (Slide.135.)

8 Here we show the setup. We ran 18 of each elec-  
9 trode each day so there were 18 positive and 18 negative.  
10 They were all run in series in one operation.

11 These are 2 liter beakers. Each beaker contains  
12 one of those impregnating jigs, as I showed. We have three  
13 hotplates to keep these solutions boiling. Here's our power  
14 supply down here.

15 This just gives an idea of the extent of our opera-  
16 tion.

17 The 4 by 4 electrode is a scale-up of about seven  
18 to one over what we had done in the previous laboratory work.

19 Next slide, please.

20 (Slide.136.)

21 Here we show the forming equipment that we used.  
22 Each electrode is formed individually in one of these cells  
23 here. We have two large-centered counterelectrodes. They are  
24 just regular centered electrodes which have a great deal more  
25 capacity than the working electrode so that-- They are charged

ab5

1 at C over 2 for four hours and then discharged and we have  
2 protection circuitry there so that the voltage can't drop below  
3 about .6 of a volt, so you discharge down to .6 of a volt.  
4 This is essentially 100 percent discharge.

5 Next slide, please.

6 (Slide 137.)

7 Here are the analytical methods we used in main-  
8 taining our solution. The nickel, cadmium and cobalt were  
9 determined by X-ray fluorescence. This is accurate to about  
10 1 percent which is perfectly good for control of this solution.  
11 It's very quick. You just dilute your solution down ten to  
12 one and put it in a little sample holder, put it in the X-ray  
13 machine and within a minute or so you have the result, what  
14 the concentration is.

15 The nitrite was determined by permanganate nitra-  
16 tion. This is a standard textbook method and is accurate to  
17 about half of a percent.

18 I used the specific ion electrode on sodium and  
19 the nitrate ion. This is not a very accurate method but  
20 fortunately these two ions-- One, we don't do anything about  
21 them anyway. In other words, if I knew they were too high or  
22 too low, I'd use it anyhow. And two, they have very little  
23 effect on the solution.

24 We've run as high as 4 molar sodium nitrate  
25 addition to our solution and find essentially no effect at all,



eb6

1 so that the only reason why you'd monitor this is that on a  
2 continuous basis, eventually you're going to build up so much  
3 sodium nitrate that you've got to do something about it. It's  
4 going to start precipitating. Actually, it's the cobalt and  
5 cadmium and nickel nitrates which will precipitate.

6 So presumably on a continuous basis you would bleed  
7 off some of your solution and precipitate out your nickel and  
8 recycle it and then I don't know what you'd do with the sodium  
9 nitrate solution. Maybe you can sell it or use it.

10 I don't know how you make sodium nitrite. Possibly  
11 you can reduce nitrate, but in any event, that will eventually  
12 have to be taken out of the solution, but we haven't done any-  
13 thing about it as yet. You can stand a very high concentration  
14 without any effect at all, and the pH was measured by glass  
15 electrode.

16 Next slide, please.

17 (Slide 138.)

18 Here we show electrochemical precipitation of cadmium  
19 hydroxide. This just gives you impregnation time versus  
20 theoretical capacity.

21 Now before anyone challenges me on the subject,  
22 theoretical capacity on either our positive or negative is  
23 determined by simply weighing the electrode and subtracting  
24 out the substrate weight. We have run experiments and others  
25 have and there is very little corrosion of the center so that--

# ANALYTICAL METHODS

ION	METHOD
Ni, Cd, Co	X-RAY-FLUORESCENCE
NO <sub>2</sub>	PERMANGANATE TITRATION
Na, NO <sub>3</sub>	SPECIFIC ION ELECTRODE
pH	GLASS ELECTRODE

Fig. 137

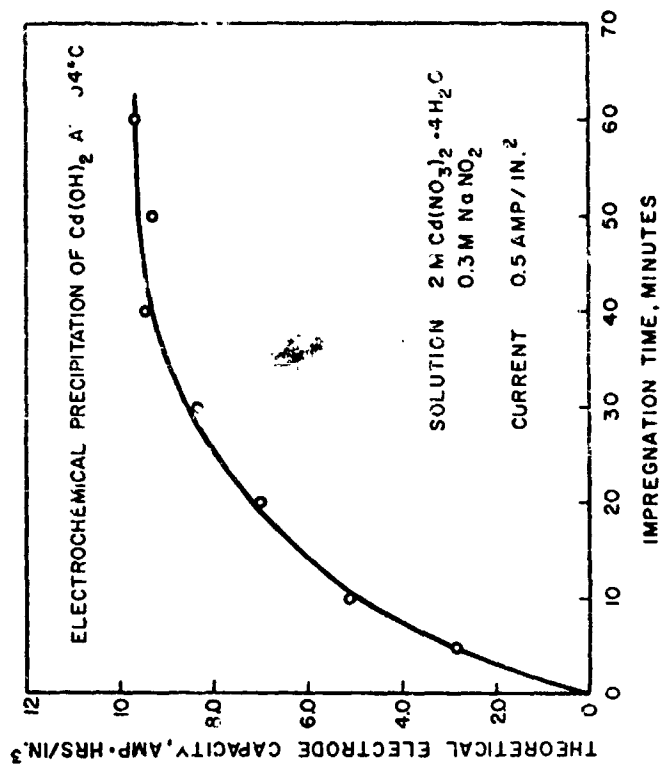


Fig. 138

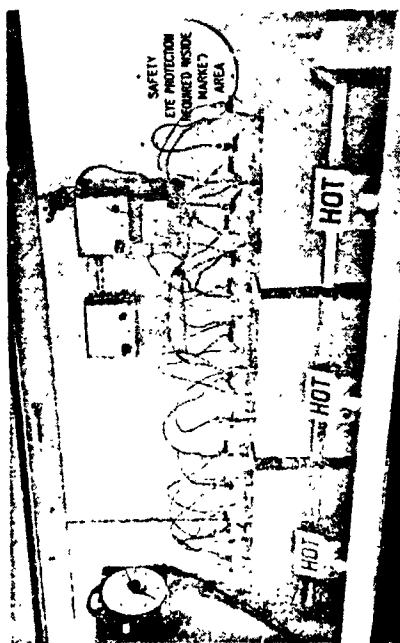


Fig. 135

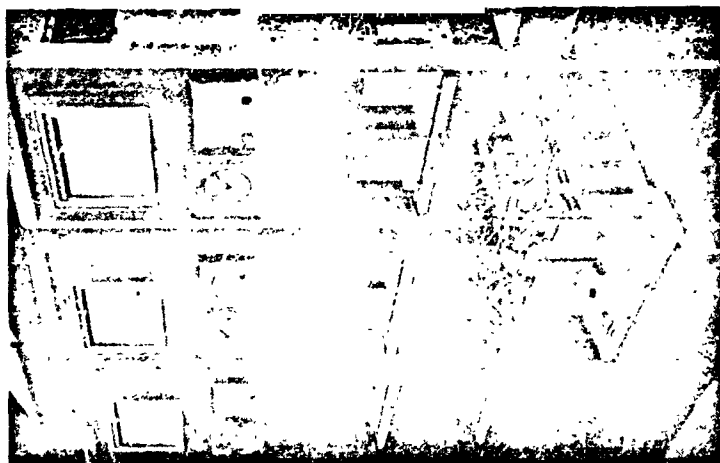


Fig. 136

eb7:

1 It's maybe one or two percent of the capacity would be corro-  
2 sion.

3 So that you can simply subtract the two numbers  
4 and get your weight of material.

5 I have assumed that cadmium goes from cadmium zero  
6 to cadmium plus plus, from cadmium to cadmium hydroxide and  
7 therefore, theoretically you should get something like .477  
8 ampere hours per gram. So this is simply the weight multiplied  
9 by a factor, and this is in ampere hours per cubic inch and  
10 if you go long enough, you can get about ten ampere hours per  
11 cubic inch.

12 We were going for a loading of 1.8 grams per cubic  
13 centimeter of void volume in the plaque which came to around  
14 six or so ampere hours per cubic inch and we did run to 25  
15 minutes on these. We stopped in the middle of the curve.

16 You could probably get more uniformity by going  
17 to the top. Then if you make a little error it doesn't matter  
18 because it isn't changing much anyway.

19 Next slide, please.

20 (Slide 139.)

21 This shows the concentration of nitrites as a  
22 function of time. We had six electrodes and we ran one for  
23 ten minutes. Well, we ran them all for ten minutes and then  
24 we kept picking one out every ten minutes and then we took the  
25 solution and analyzed the nitrate.

eb8  
1 The rate of loss of nitrite is a little higher in  
2 the cadmium solution but I think this is because you have not  
3 only electrochemical decomposition of nitrites but you also have  
4 a chemical decomposition. Nitrite is somewhat unstable,  
5 especially in a hot, slightly acid solution.

6 So the higher the concentration, the higher will be  
7 the rate of loss. So one would expect that the two lines would  
8 be -- the cadmium would have a slightly higher slope to it.  
9 And you see at the end of the run in the nickel we're getting  
10 rather thin in nitrite. In the cadmium we still have suffi-  
11 cient.

12 Next slide, please.

13 (Slide-140.)

14 This is an electrode, a 4 by 4 electrode which was  
15 cut into 1-inch squares. The tab was up in that corner there  
16 and we kind of figured that particular square doesn't count for  
17 much because I have a tab on it. You add the weight of the  
18 tab but then you subtract the weight of what you would have  
19 impregnated there. Actually, this should be discounted alto-  
20 gether because by chance it comes out about the same as the  
21 other.

22 But what I did was weigh all the electrodes, get  
23 the average, all these little squares, get the average and  
24 then I calculated deviations and this is simply a percent.  
25 This one was 1.4 percent higher than the average, and this is

eb9. 1 .6 percent lower.

2 But we see that the deviation from highest to low-  
3 est is plus or minus 2.5 percent and this includes the error  
4 that you would get from the plaque itself. They are within  
5 about a percent, and then the error that you get from me cutting  
6 the thing. I have to cut it in little shears and if I miss  
7 by a hundredth of an inch, that's 1 percent.

8 So essentially this thing is quite uniform across  
9 the whole area of the plaque. This is an impregnated plaque.

10 Next slide, please.

11 (Slide 141.)

12 We then took one of the 4 by 4 electrodes and we  
13 cut it up into little squares and put little tabs on them and  
14 ran them as four 1-1/2-inch electrodes. That comes to about  
15 half the area of the original plaque but the 1-1/2-inch squares  
16 were cut out of the four corners, and these were cycled at a  
17 very high rate. I think it was 10 C rate.

18 As you see, they are quite uniform and they all  
19 tend to go out together. They're within 10 percent of each  
20 other in the capacity over -- which comes to -- let's see, at  
21 two, three, about four hundred cycles they begin to bend down.  
22 Well, they're only down about maybe 20 percent at the end of  
23 the thousand cycles.

24 Next slide please.

25 That's all there is? Okay.

ebl0

1 I have here-- This is a Vu-graph.

2 (Slide 142.)

3 This is a summary of the data that we gathered over  
4 essentially 300 positive and 300 negative electrodes. We find  
5 a theoretical capacity in the cadmium of 4. -- No, that one is  
6 a mistake. This is about 3.219. It's about a 74 percent  
7 utilization on that.

8 This is what you get for having these things typed  
9 up the minute before you go.

10 The standard deviation is about 5 percent on this  
11 large number of electrodes. If you take one string of 18--  
12 They're made in strings of 18 -- the better ones will run down  
13 to a little less than 2 percent standard deviation on that  
14 group.

15 The nickels will run in two groups. We had one  
16 battery with a little bit higher capacity than the other, so  
17 we increased our-- We were running a 30-minute deposition and  
18 a 40-minute deposition, and so we get a higher capacity in the  
19 second group.

20 Here we're getting I think pretty good standard  
21 deviation on these electrodes of essentially three, and in the  
22 measured capacities we're getting approximately, well, 4 to 5  
23 percent standard deviation. So it's quite a uniform process.  
24 This considers a whole bunch of electrodes made on a semi-  
25 production scale sort of, and we do have a higher measured

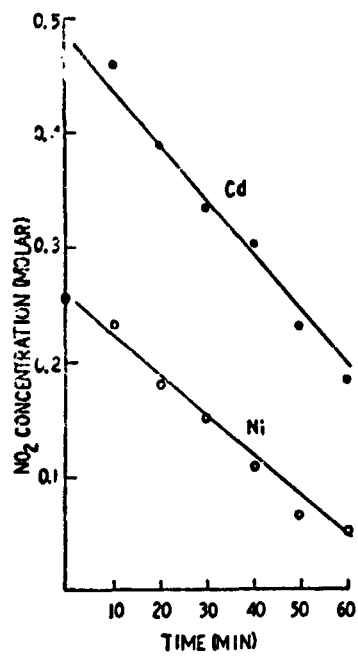


Fig. 139

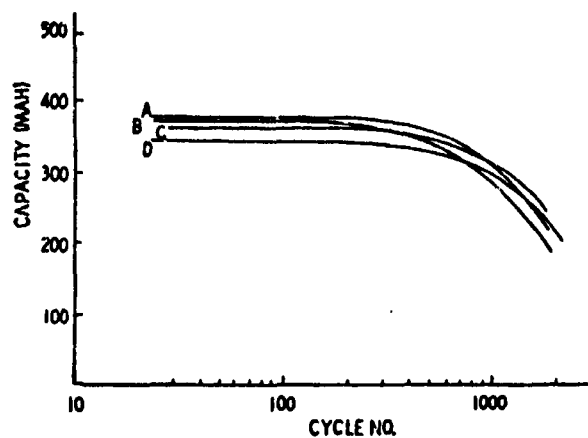


Fig. 140

# ELECTRODE WEIGHT DISTRIBUTION % DEVIATION FROM MEAN

+1.4	-1.3	+0.3	-2.5
+2.1	+1.6	+2.8	-0.6
+2.0	-0.3	+0.7	-0.4
-0.7	-0.3	+1.9	-2.5

Fig. 141

# ELECTRODE PERFORMANCE AH CAPACITY AT C/2

	Cd	Ni	
		Group 1	Group 2
<u>Theoretical</u>			
Mean	4.401	1.613	1.786
SD	0.219	0.046	0.052
%	5.0	2.7	3.1
<u>Measured</u>			
Mean	3.287	1.935	2.052
SD	0.204	0.104	0.069
%	6.2	5.4	3.3

Fig. 142

eb11 1 capacity than theoretical and this is explained by saying that  
2 the reaction is not a one-electron transfer.

3 In other words, if you overcharge a nickel electrode  
4 you get more capacity than would be dictated by a nickel 2 to  
5 nickel 3 oxidation.

6 And this is essentially a conglomerate of all the  
7 electrodes we got and it shows I think a fairly good reliability.  
8 I don't have these things as a function of time. There was no  
9 degradation with time in our solution. As the solution aged  
10 there was no variation with aging.

11 We had had troubles in the past when we were using  
12 a polyvinyl chloride storage container. We leached some kind  
13 of garbage out of there which had a tendency to slowly poison  
14 the cadmium solution for some reason. So we have stuck to  
15 glass, teflon, platinum and titanium in this work.

16 We are working on other materials to see if we could  
17 get away perhaps with a 410 stainless but-- For economic  
18 reasons we would like to do this but for practical reasons we  
19 don't want to use anything we are not sure of at the moment.  
20 We have to test out the other material.

21 \* And that's the whole story.

22 HALPERT: Thank you, Ed.

23 Any questions for Ed?

24 O'ROURKE: Joe O'Rourke, Grumman.

25 In that slide you had, the checkerboard type one



eb12

1 where you had weight distribution of each one, you talk about  
2 the weight deviation per square, about a mean. Now is that the  
3 mean of each square? Is that the average deviation, about the  
4 average weight of each square, or about the weight of the whole  
5 plaque?

6 MC HENRY: I just took the average weight and then  
7 took the percentage deviation of each square from that average  
8 weight, and then that is a real number on each, and the spread  
9 is not a standard deviation. Plus or minus is the range; it's  
10 not a standard deviation.

11 The standard deviation I suppose would be about a  
12 half -- it would probably be plus or minus 1-1/2, something  
13 like that.

14 HALPERT: Thank you, Ed.

15 We'll continue with more discussion on the Bell  
16 process. Dean Maurer is going to talk about sealed nickel-  
17 cadmium cell design for Bell systems service.

xzxzx 18 MAURER: Yesterday you heard quite a variety of  
19 methods of charging control, all based on quite a lot of  
20 electronic circuitry. There was pulse charging and there was  
21 PPC's and PCC methods. What I would like to present here now  
22 is a minority report, a charging control method that we use  
23 which we'll call ZCC, for zero charge control.

24 (Laughter.)

25 It requires the use of a resistor and a diode and

eb13 1 you simply charge continuously and the major difference is  
2 that you get a lot of overcharge as opposed to a very limited  
3 amount.

4           The problem with this type of method is that it  
5 requires a cell with some peculiar design features which I will  
6 talk about.

7           (Slide 143.)

8           The performance is something of this sort, in which  
9 we've plotted the measured capacity to one volt versus the  
10 charging current ranging from C over 20 to the 2 C rate, giving  
11 these capacities and pressures shown on the lower curve.

12           In the right-hand axis you will notice that at 2  
13 C, we're somewhere around seven pounds absolute pressure, not  
14 gauge.

15           And to give you the callibration, since everyone  
16 who talks uses a different set of units, we have the theoretical  
17 capacity of the positive electrodes, again based on one electron  
18 transfer shown here, so that we are above theoretical capacity  
19 when we charge at rates higher than about C over 5 or so.

20           This theoretical line, in terms that Steve Gaston  
21 used yesterday, would correspond to the 267 milliamperes hours  
22 per gram of active material -- 287, rather, at this point, so  
23 these points are up around 340 milliamperes hours per gram of  
24 active material, just as a cross-calibration.

25           So that now we can design cells which can be

eb14 1 rapid charged at the 2 C rate if we like without worrying about  
2 overcharge. We can turn them off on the basis of a thermal  
3 sensor if we like, just to keep the rest of the package from  
4 getting too hot.

5 The way one designs cells like this--

6 (Slide 144.)

7 Now before I go further, these are the two cell  
8 designs that I'll be talking about. This one is a cell whose  
9 performance I just showed. It's about 100 mills thick and it  
10 has two modified Ziegler seals on it; it's a prismatic design.

11 This is a 23 ampere hour design, again with Ziegler  
12 seals.

13 The key to the performance of a cell on continuous  
14 overcharge, either in reserve use in terrestrial environments  
15 which are quite severe, temperatures from typically minus 20  
16 Fahrenheit to plus 140 Fahrenheit, and also the problems of  
17 making cells capable of rapid recharge all boils down to the  
18 same point: You want to keep the pressure down, whether it's the  
19 oxygen pressure during rapid recharge or whether it is hydrogen  
20 or the lack of hydrogen pressure, during low temperature  
21 operation especially, or the accumulation of hydrogen on life,  
22 and it is all related to the negative electrode.

23 First I'll consider the oxygen problem.

24 If you plot the oxygen pressure, the equilibrium  
25 pressure, versus the negative loading --

eb15

(Slide 145.)

-- I've plotted it here as percent of the negative plaque volume that's filled, but you could consider it a variety of other ways if you like.

But you get a curve for different levels of electrolyte that break upwards fairly sharply. The usual negative loadings that you see in commercial cells tends to be up toward the 40 percent region and consequently the pressures are relatively higher and sensitive to the volume of the electrolyte in the cell, a relatively unstable type of a region. And we feel that the best place to operate is down near the knee of this curve where there is little variation in pressure with electrolyte quantity and one can get the theoretical capacity out of the positive and still maintain low pressure.

And of course things like state of charge will affect these curves. In essence, as you increase the state of charge on the negative you make it more porous, so a given curve will move further towards the right for a given loading; when you increase the state of charge you'll get a lower pressure.

And again, down in this region it won't make very much difference, but if you're up in this region, out in this area, it will make a substantial difference.

What this means then is if you plot the oxygen pressure versus the charge rate, -- there's a log charge rate

(Slide 145A.)

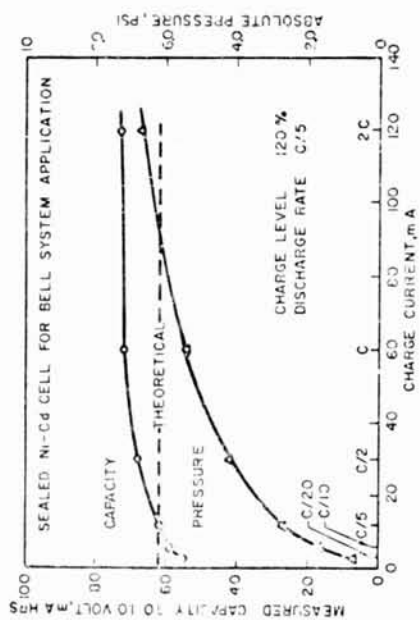


Fig. 143

EFFECT OF NEGATIVE LOADING AND QUANTITY OF ELECTROLYTE ON STEADY STATE OXYGEN PRESSURE

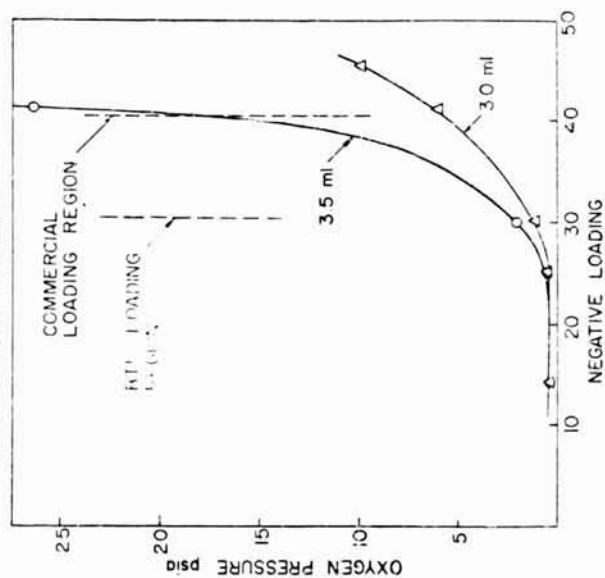


Fig. 145

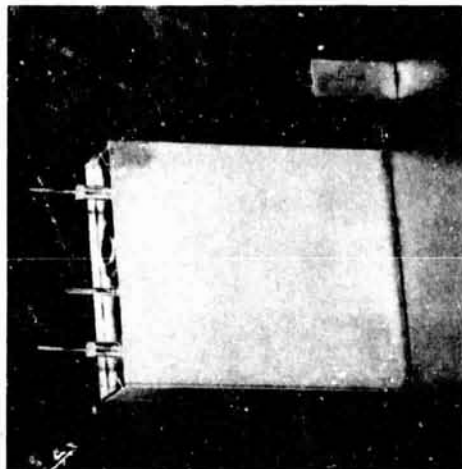


Fig. 144

EFFECT OF CHARGE RATE ON STEADY STATE OXYGEN PRESSURE

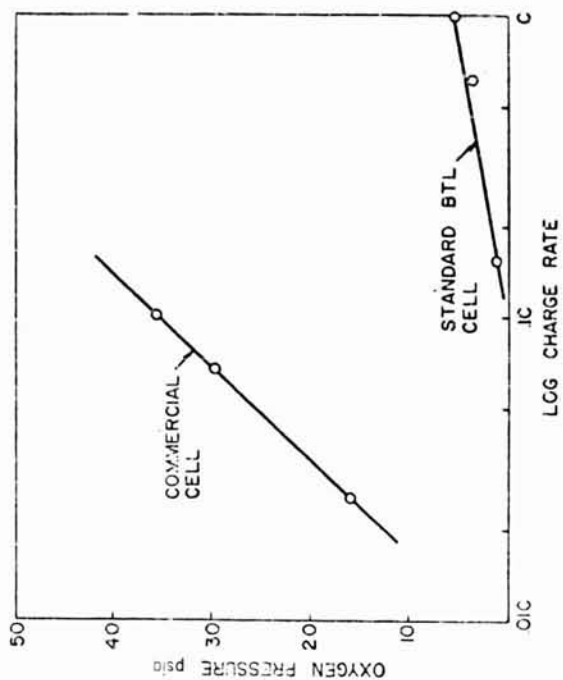


Fig. 145A

eb16

1 and a 10-hour and 1-hour rate -- cells operating down in the  
2 30 percent fill region give you very low oxygen pressures where-  
3 as those operating up in the 40 percent region give relatively  
4 higher pressures.

5 So now, using just this consideration, you can make  
6 a cell then that can be recharged -- charged continuously at  
7 the 30-minute rate.

8 (Slide 146.)

9 Now let's talk about the negative electrodes.

10 Looking at the negative polarization versus the  
11 charge return on a negative electrode you get a typical curve  
12 looking like this: a constant potential region where the nega-  
13 tive is charging. This then breaks to a higher potential  
14 region where you get hydrogen evolution, and if this were a  
15 sealed cell or if oxygen were present, then the oxygen de-  
16 polarizes the negative and the voltage drops to a lower level,  
17 you do not get hydrogen evolution and in fact further charging  
18 of the negative ceases.

19 So one normally then designs a cell with a positive  
20 lower capacity than the negative, as you all know, and you get  
21 the oxygen coming over before the negative becomes fully charged  
22 and things are great.

23 Now if you look at these same curves at either  
24 higher rates or lower temperatures or both, the curve shifts  
25 like this. The charging region comes up in voltage. There is

(Slide 146A.)

eb17

1 an initial high voltage on a charging curve in which you get  
2 some hydrogen evolution and the point of hydrogen evolution  
3 toward the end of the charge has moved substantially to the  
4 left at the lower temperature and/or higher rate.

5 And just where this shifts to depends first on  
6 the conditions you're talking about and also the previous  
7 condition or history of the electrode.

8 So then what you want to do to design a cell is  
9 arrange to have the positive capacity operate within this band  
10 between the preliminary hydrogen evolution and the final  
11 hydrogen evolution region.

12 I've marked here-- This is the range of the safe  
13 positive theoretical capacity if you have electrodes which  
14 generally give you less than theoretical capacity but as we  
15 were discussing yesterday, some cells which are continuously  
16 charged, especially at lower temperatures, give higher and  
17 higher capacities and in fact we've found in most cases this  
18 approaches the theoretical capacity of the electrode, especially  
19 around zero degrees Centigrade.

20 In the positive electrodes that we make by the  
21 electrochemical process this has to be 120 percent of the  
22 theoretical capacity, something in that vicinity.

23 Now one can plot-- For a given charging tempera-  
24 ture one can plot the hydrogen free capacity, that is, the  
25 capacity in before this hydrogen evolution ever occurs,

eb18

(Slide 147.)

1  
2 -- and that looks like this, on which I've plotted the hydrogen  
3 free charge return in terms of percent of the rated capacity  
4 of the electrode versus the charge rate in terms of percent  
5 rated capacity, and looking at it for different preconditioning  
6 cycles and different charging temperatures.

7           So that here are a set of data in which the  
8 electrode was equilibrated at 77 degrees F. at the five-hour  
9 rate first. Then it was lowered in the discharge phase to  
10 zero and charged at one of these rates, and we generate a  
11 curve that looks like this.

12           Now if in fact we had gone to a lower temperature  
13 than zero, minus 20 for instance gives a curve that has dropped  
14 all the way down to this region. If, on the other hand, the  
15 electrode is equilibrated at 140 Fahrenheit first and then  
16 lowered to zero, the curve shifts from this position to this  
17 position.

18           Now these are not such outlandish conditions as you  
19 might think at first off. Zero degrees followed by equilibra-  
20 tion at 140 is simply the cold winter following a lot hot  
21 summer in a piece of pole-mounted telephone equipment. The  
22 equipment is in a box in the hot sun. The temperature gets up  
23 in the 120, 140, even 160 region. And in the winter of course  
24 it drops.

25           So that if one does not have enough excess negative



eb19

1 capacity to keep out of this hydrogen region, you will develop  
2 hydrogen gas and the cell will vent.

3 Also, for a reserve battery with perhaps a two to  
4 one negative to positive ratio, at the 20 hour rate of charge,  
5 that's a 40 hour rate on a negative so one is talking about  
6 something down here very close to the axis, so these effects are  
7 not all that drastic but they are present.

8 If you wanted to design a cell for rapid recharge,  
9 however, you have to take these into account and have quite a  
10 substantial excess capacity for rapid charge at very low tem-  
11 peratures.

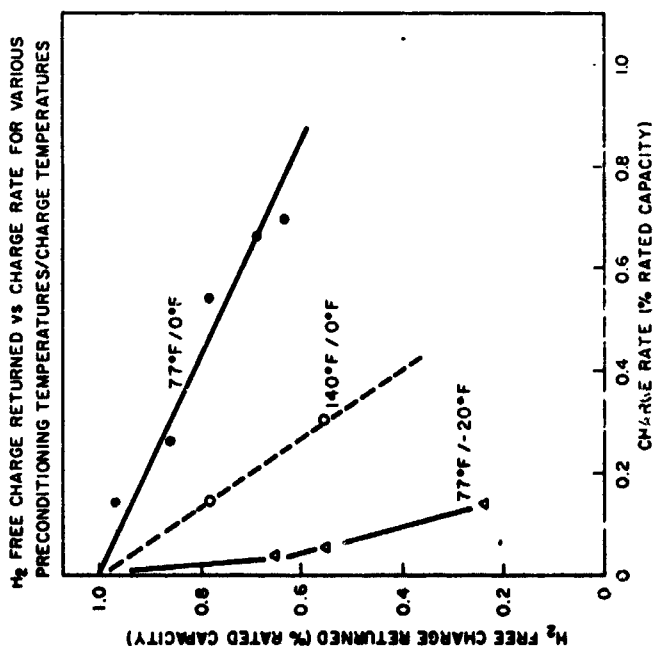
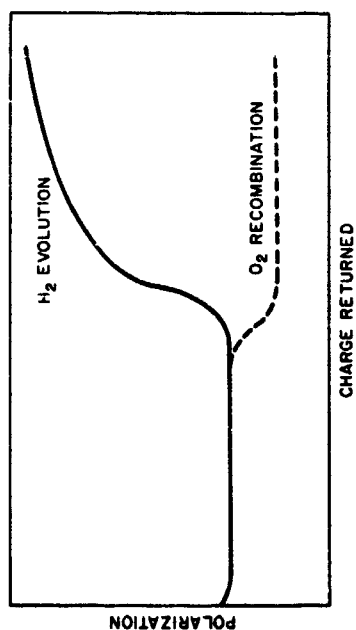
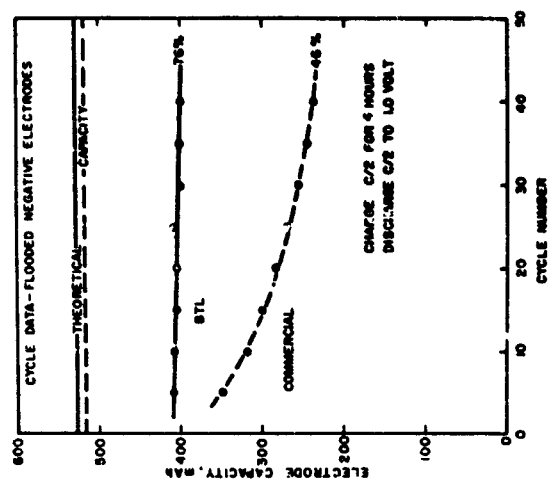
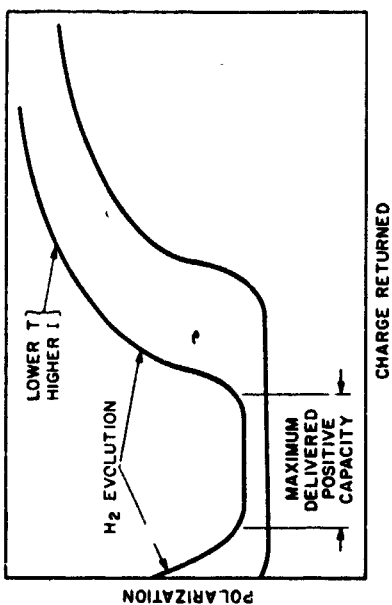
12 Now that takes care of the time zero negative condi-  
13 tion. The negative doesn't stay in that state throughout its  
14 life. You have to worry about some other things happening,  
15 fading, for example.

16 (Slide.148.)

17 These were shown last year, the theoretical capacity  
18 of the electrode and the electrochemical electrode, and here  
19 a particularly -- a rather bad commercial electrode with a  
20 fairly sharp drop with cycling in capacity.

21 Now this electrode-- A cell designed on the basis  
22 of that electrode would have to have much larger ratios in  
23 order to keep you out of trouble later in life.

24 Now I want to emphasize that cycling in the flooded  
25 state on negatives is not the same as cycling in sealed state,



eb20-

1 and just what the relationship is I don't know, but one gets  
2 longer life, cycle life on negatives in the sealed state.  
3 Part of this of course is due to the fact that you don't cycle  
4 them through nearly as great a range in the sealed state.

5 Now while this material is electrochemically in-  
6 active it is still present as a chemical species within the  
7 sealed cell and has to be contended with, and we propose the  
8 following things going on.

9 (Slide.149.)

10 First, we represent the positive battery by this  
11 block, the negative by this block. It's divided up into the  
12 black area representing the electrochemically active material  
13 and the red area representing the inactive material which I  
14 think you will generally agree is usually cadmium that's  
15 isolated, and most chemical analyses show this to be cadmium  
16 metal and not hydroxide.

17 Then this is divided up into the working capacity  
18 which matches the positive, some excess negative cadmium hydro-  
19 xide to account for the low temperature charging. You have an  
20 amount here now-- You design the cell to have an amount there  
21 that matches -- to give you the hydrogen free performance for  
22 the highest rate and the lowest temperature you intend to design  
23 the cell for.

24 You have a certain amount of precharge material  
25 and basically all that we're concerned with in the design is

cb21

1 the precharge to keep you out of that preliminary hydrogen  
2 evolution region that I showed earlier. Then some things can  
3 happen.

4 (Slide-150.)

5 On overcharge, oxygen in the cell can oxidize this  
6 cadmium metal. It is not isolated chemically, just electro-  
7 chemically. So the cadmium plus the oxygen and water will give  
8 you some cadmium hydroxide but since the other half of the  
9 electrochemical couple has to be satisfied, some cadmium hydro-  
10 xide gets reduced at the same time.

11 So one transfers active cadmium hydroxide to active  
12 cadmium in exchange for inactive cadmium to something that  
13 I'll admit is questionable. This can be either active or in-  
14 active, but I would assume that it's quite a bit inactive,  
15 let's say.

16 So one needs to design for this and allow enough  
17 extra cadmium hydroxide present on the negative to match the  
18 amount of inactive material that you have because it is poten-  
19 tially capable of being transferred to active cadmium at the  
20 expense of cadmium hydroxide which you need for the low tempera-  
21 ture operation.

22 So then what this says then is that you have the  
23 over-all design of the cell now. You have the working cadmium  
24 capacity which is equal to the requirements of the cell design.

25 If you want a 12 ampere hour cell you have a 12 ampere hour

eb22

1 working cadmium capacity.

2           Then you make the electrode some maximum thickness,  
3 probably around 30 mills, and you load it to the 30 percent  
4 point so this gives you the amount of cadmium area, cadmium  
5 electrode area that you require-- I'm sorry, you add enough  
6 cadmium precharge and hydroxide to account for the effects that  
7 I've been talking about.

8           Then you match this total area with a positive  
9 electrode. That usually winds up to be a very thin electrode  
10 because you don't need very much capacity per square inch to  
11 match the cadmium. Then you make the positive loading as high  
12 as you can to maximize the energy density of the cell.

13           We've made the cells in this form. I showed you  
14 the data on the small cell initially, capable of rapid re-  
15 charge.

16           (Slide .151.)

17           Here are some data on some -- on one of our cells  
18 on continuous overcharge. Here we have a C over 20 overcharge  
19 over 20 degrees Fahrenheit but these cells had been previously  
20 conditioned 35 days at 110 Fahrenheit on overcharge, so again  
21 this is the winter following the hot summer.

22           Now I plotted pressure in pounds per square inch  
23 absolute versus time and the cell has shown low stable capacity.  
24 And for comparison I have three different other cell designs  
25 on the same plot that were not designed to take all these

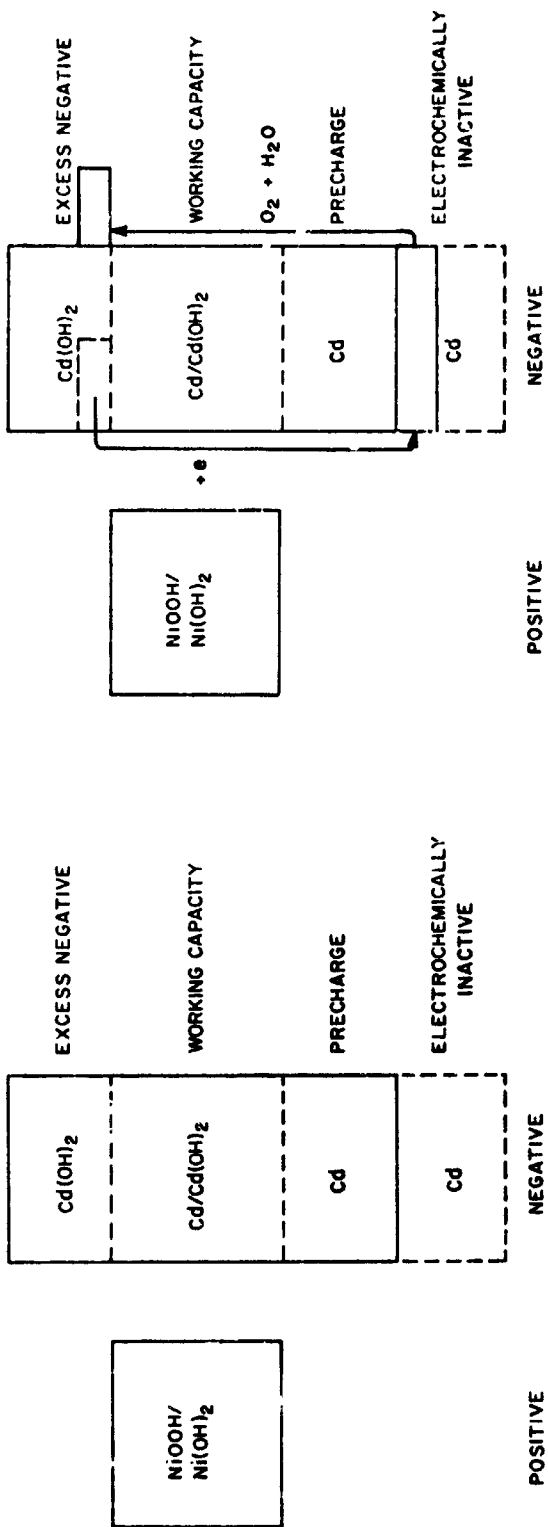


Fig. 149

Fig. 150

Pressure on C/20 Overcharge at 20°F  
Following 35 days 110°F

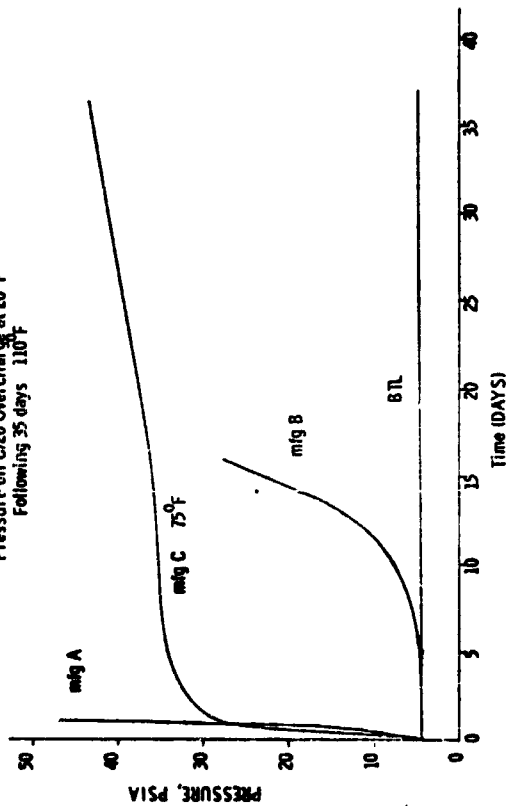


Fig. 151

523 1 factors into account.

2 One of these fell out right at the start. Another  
3 one went for a while but then started to generate hydrogen,  
4 presumably because of the exchange effect that I spoke of.

5 And another guy that really doesn't belong on this  
6 plot per se. At 75 Fahrenheit it started to generate hydrogen  
7 after several weeks on overcharge.

8 So that by taking all these into account, one can  
9 design cells that can be used on continuous overcharge. I  
10 think that the aerospace industry might consider some of these  
11 facts in their cell design, especially the effects of over-  
12 charge on the electrode performances and perhaps opt for a  
13 little more overcharge at least than is presently being used.

14 Thank you.

15 HALPERT: Are there any questions for Dean? Any  
16 comments?

17 FORD: Ford, NASA-Goddard.

18 Dean, would you care to comment on-- You mentioned  
19 how the hydrogen free capacity changed with life. Have you had  
20 any experience or observed any difference in cell potential  
21 through which you can get hydrogen as a function of life at  
22 these steady or these constant current overcharge rates?

23 MAURER: No, I don't think I could quote numbers  
24 off the top of my head on that specific thing. We have observed  
25 voltages and you can get hydrogen evolution to vary over quite

eb24

1 a wide range from safe operation above 1.6 to hydrogen genera-  
2 tion at 1.55.

3 Of course we have observed hydrogen generation as  
4 I pointed out here even at room temperature even though the  
5 voltages were not very high. As I remember it, the voltage was  
6 probably in the range of 1.45.

7 FORD: . . . Gerry, do you have another question here?

8 DUNLOP: Jim Dunlop, Comsat.

9 Do you have any observed mechanism for hydrogen  
10 recombination once you get it generated?

11 MAURER: No, but I'd like to have some.

12 (Laughter.)

13 LACKNER: Lackner, Canadian Defense Research.

14 On the hydrogen free charge return, are we talking  
15 of negative-to-positive plate balance or ratio? You haven't  
16 given us a figure for that.

17 MAURER: What we finally used?

18 LACKNER: Right.

19 MAURER: We use a ratio of theoretical positive  
20 capacity to measured negative capacity of 2.2 to one-- I'm  
21 sorry, negative to positive of 2 to one. And that, in theoret-  
22 ical terms, theoretical negative to theoretical positive, is  
23 2-1/2 to one.

24 LACKNER: The other question is facetious. If  
25 your precharge, which I think we're on record we don't like,



eb25 1 prevents the onset of hydrogen evolution, why don't you have a  
2 larger percent of precharge so you have a one-to-one ratio?

3 MAURER: All you need is enough precharge to get  
4 out of that initial region, and that only amounts to something  
5 like 10 percent of the negative capacity. This depends on the  
6 particular electrode that we're talking about. All you need to  
7 do is get out of this region and that in general is perhaps  
8 only 10 percent of the negative capacity so you just put in  
9 enough to get you out of that area.

10 LACKNER: Does that mean that if you started with  
11 a totally discharged negative and a totally discharged positive,  
12 you would evolve hydrogen at the beginning?

13 MAURER: It would if you did it at a low temperature  
14 and a high enough rate.

15 LACKNER: At normal rates of, say, C over 20, C  
16 over 10 at minus 5, would you evolve hydrogen?

17 MAURER: Again, it depends on the electrode and  
18 its preconditioning. I would guess that if you would equili-  
19 brate it at 140 degrees first, and you're talking about minus  
20 5 and C over 10 on the negative capacity then you might very  
21 well get a burst of hydrogen.

22 LACKNER: You're making a generalization and you're  
23 taking specific cases here of preconditioning electrodes and  
24 what-have-you. As a general electrochemical phenomena, you  
25 don't evolve hydrogen from the negative until you get into the

e526

1 overcharge region, so I'm asking:

2 If you start with a fully discharged negative and  
3 a fully discharged positive, what causes hydrogen to be evolved?

4 MAURER: You get hydrogen evolved-- On the  
5 negative process there is in general three polarizations to  
6 concern yourself with. One is the polarization for hydrogen  
7 evolution on nickel surfaces; another, the polarization --  
8 activation polarization for cadmium reduction; and the polari-  
9 zation -- concentration polarization; and the polarization for  
10 hydrogen evolution on cadmium.

11 Now on the very start of the charge on a completely  
12 discharged electrode there are exposed nickel sites available  
13 and one can have a condition in which the hydrogen polarization  
14 on nickel takes over and you get some hydrogen off before  
15 cadmium covers these areas during part of the charging process  
16 on cadmium. So it's a burst of hydrogen; it's not a continuous  
17 evolution that continues throughout the cell charging.

18 But if you're talking about cycling the cell under  
19 those conditions, then you can accumulate some hydrogen, but  
20 just where the process occurs depends on each particular cell  
21 design and all I'm saying is that one needs to take all of  
22 these factors into account in designing the cell.

23 Maybe for your particular application, your parti-  
24 cular conditions, this won't be a problem that you have to  
25 worry about at all. In our set of conditions we have to worry

ab27

1 about it a little bit.

2 LACKNER: It's been our experience that the less  
3 precharge you have the better off you are, but it's hard to  
4 convince anybody else.

5 MAURER: I agree with that in the sense that you  
6 want the excess cadmium in the form of cadmium hydroxide so  
7 that the ratio doesn't have to be any higher than you absolutely  
8 need, because the higher the ratio the lower the energy density  
9 becomes and so on.

10 I don't think you need precharge for the purpose  
11 that many people put it in, namely to lower their oxygen pres-  
12 sure. I don't think it's especially necessary for that. If  
13 you load the electrode low enough you can have low enough  
14 oxygen pressure.

15 And the other reason that some people put it in  
16 is because of fading of the negative on some types of cycling  
17 regimes. I think that's accentuated when you have very limited  
18 or no overcharge. Then one tends to get into negative fading  
19 more -- in a more pronounced way than you would if you had  
20 overcharging.

21 STEINHAUER: Steinhauer, Hughes.

22 Dean, would you say then-- I noticed you ran a  
23 long-term 35-day overcharge test-- that if you don't have the  
24 opportunity to have fully instrumented cells, say for gas  
25 partitioning or for pressure, would a long-term low temperature

eb28

1 overcharge test be a reasonable indication of adequate over-  
2 charge protection or cadmium hydroxide upstairs, along with  
3 voltage?

4 MAURER: No, not just in itself. You need the over-  
5 charge. You also need something in the way of preconditioning  
6 to the worst case condition. And then you have to have some  
7 idea how the negative might be fading in capacity with life  
8 and account for that as well.

9 Because what you want, you want to eliminate all of  
10 these factors as failure modes so that what you do is look at  
11 what the negative is going to be looking like far out in time  
12 and allow for enough excess capacity and enough excess cadmium  
13 hydroxide way out in time so that you won't get into trouble.

14 Otherwise, this might very well become the failure  
15 mode itself.

16 HAINES: Haines, Defense Research Establishment,  
17 Ottawa.

18 I'd like to come back to this hydrogen evolution.  
19 If I understand what you said correctly, it's when you have your  
20 negatives completely discharged, you have nickel sites where  
21 you can possibly get the evolution of hydrogen.

22 Now are you stating that when the thing is partially  
23 charged, these nickel sites are covered? And if they are not,  
24 then how come you don't get hydrogen evolution every time you  
25 start to charge?

eb29

1 MAURER: Well, I think it's a matter of degree.  
2 I think that once you've started to charge you cover enough  
3 of them that you get out of the problem to some extent.

4 HAINES: I just can't see how the charge cadmium  
5 will cover these nickel sites.

6 MAURER: Well, the charging action goes through a  
7 soluble intermediate and the cadmium will sit down on any  
8 electrically active site available, and there are probably more  
9 nickel sites available initially than anything else.

10 In fact, if you can think of a pristine state,  
11 and I'm not sure you ever really have this, depending on the  
12 method of impregnating, where you have cadmium hydroxide pre-  
13 sumably throughout most of the -- covering much of the area  
14 in the cell and nickel -- there isn't any cadmium initially --  
15 and then as that becomes reduced it has to go down on some  
16 substrate, conductive substrate within the electrode.

17 GINER: Jose Giner, Tyco Laboratories.

18 I think there probably may be another explanation, and  
19 I want to suggest a look at the old papers of reduction of  
20 oxides by Ohse and Nagel and many other people have shown  
21 that when you have a completely oxidized substance like cadmium  
22 oxide or silver oxide, you have always an overshoot of potential  
23 to reduce the beginning because you have to create some  
24 nucleation for the further reduction.

25 So it is possible that if you have only cadmium

MV-10

1 oxide the potential overshoots in order to create these.

2 So, in the overshoot you may evolve hydrogen.

3 Therefore, I think that probably you want to have  
4 the cadmium, free cadmium, to avoid the potential overshoot  
5 that you get when you nucleate.

6 MAURER: Yes. So that you are saying that, instead  
7 of nickel sites per se, you now have some cadmium sites as  
8 well?

9 GINER: You need them for the cadmium oxide reduc-  
10 tion.

11 DUNLOP: I want to comment on that, too. This over-  
12 shoot that you look at, you normally observe when you completely  
13 discharge the cell for a long period of time and that's the  
14 reason why almost all the battery manufacturers recommend that,  
15 if you have cells that are shorted in your laboratories for a  
16 long period of time, that you exercise a great deal of caution  
17 when you initially start charging.

18 Now, after you've cycled the cells for a while, it  
19 seems that you don't really have this problem, and our results  
20 would indicate the reason you don't have this problem is that  
21 you now have more actively charged cadmium available to you.

22 MAURER: That's true, at room temperature.

23 GINER: At room temperature.

24

25

eb31

1           The other point that we have observed in the cells  
2 that are being made for us using the normal SAFT process is  
3 that when they make the cell initially they're going to have  
4 this 10 percent charge cadmium in there because they can't  
5 get it out of there. It's there, and there is no way initially  
6 to discharge it after the cell plates come out of the forma-  
7 tion cycle.

8           So it's really not something you have to put in;  
9 it's really in there anyway, and they really can't, unless they  
10 change the procedure, the current procedure, they can't get  
11 it out anyway.

12           MAURER: What you say is partially true and would  
13 be true at room temperature operation but if you take those  
14 electrodes that have been completely discharged electrochemically  
15 at room temperature and dropped to low temperatures, you get  
16 this effect again anyway. So that amount of precharge isn't  
17 enough to account for, let's say, 20 degree or zero degree  
18 operations.

19           SEIGER: Seiger, of Heliotek.

20           Dean, in the first part of-- During that period  
21 of hydrogen evolution, what's happening to the temperature of  
22 the cell? Is it getting very hot, or is it staying moderately  
23 cold?

24           MAURER: It's moderately cold.

          HALLECK: Halleck, Tyco Laboratories.

eb32

1 I have a question pertaining to the fade of the cad-  
2 mium electrodes. Do you know anything about whether it  
3 fades faster or less fast if you have a high rate, a continuous  
4 high rate of overcharge?

5 MAURER: No. I have a few generalizations,  
6 that's all they are at the moment. We're going to do some of  
7 these studies, look at the effect of overcharge and rate or  
8 lack of overcharge at all on the negative.

9 But in general, I think the higher the rate, the  
10 worse the fading is.

11 HALLECK: You mean the higher the rate of over-  
12 charge?

13 MAURER: The higher the rate of discharge, the more  
14 fading one accumulates. As far as charging, I'm not sure.  
15 I don't have any real feeling for that.

16 FORD: Ford, NASA/Goddard.

17 I'd like to make two comments, one in regard to  
18 Bob Steinhauer's point on the low temperature overcharge test  
19 for long periods. It is my belief that that only is a status  
20 indicator of the present condition of the cell and as Dean  
21 indicated, that will change on a day-to-day basis.

22 But I think we are missing the major point here.  
23 Dean showed a curve labelled "commercial plates" of the degrada-  
24 tion in ampere hour capacity of the negative electrode. I  
25 can show you data in NASA reports of commercial aerospace



eb33

1 plates that look essentially the same. So you might draw your  
2 own conclusions from that.

3 But my point is simply this:

4 I think we are basing negative capacity total in  
5 a cell -- this is measurable capacity as required in the speci-  
6 fication, the ratio test, and parity capacity. We are basing  
7 our ratio on an early point in life. We are not taking into  
8 consideration this degradation, and I am proposing today, and  
9 I think you will see it very shortly in future NASA specs,  
10 that one of the requirements for qualifying a group of nega-  
11 tive electrodes for a cell is to run the cycle test to demon-  
12 strate capacity stability, and I think this is something we  
13 should be looking at very hard.

14 HALPERT: Any other comments on this point?

15 One more.

16 THORNELL: Thornell, ESB.

17 What are the energy densities on these cells?

18 MAURER: I'm not sure what it is for the 23. For  
19 the little one it's in excess of one watt hour per cubic inch.  
20 I don't know what it is in terms of weight.

21 HALPERT: Okay.

22 We're going to start our pre-charge session. I  
23 guess we're going to have to do that directly after lunch.

24 We have one more paper for this morning, and that is by Will  
25 Scott, who wants to present some analysis data from the state

eB34 1 of the art plate materials.

xzxzxx 2 SCOTT: I'd like to present some data that has been  
3 generated to date on a program that is supported by in-house  
4 resources at TRW directed toward determining the fundamental  
5 limitations to operating cells at very high utilization factors  
6 or very high depths of discharge, whatever you wish.

7 The orientation of the work is toward applications  
8 at the moment in synchronous equatorial orbits where one has  
9 the freedom at least, let's say, of choice because of a rela-  
10 tively low number of cycles, at least compared to what is re-  
11 quired in a low earth orbit.

12 This work is in support of, at the moment, of  
13 various other hardware and development programs at TRW directed  
14 toward long-term synchronous orbit type applications.

15 Now most of you have seen various kinds of analyti-  
16 cal data for plate material in the past and I'm not going to  
17 show you, I hope, the same kind of data that you may have seen  
18 before. What I would like to show you is some data for some  
19 plate material made under a special set of conditions but not  
20 necessarily any different basic process conditions than is  
21 ordinarily being used today.

22 The main difference is that the material was -- the  
23 data that I'll show you today was taken from plates all taken  
24 from a single spiral during the production cycle and therefore,  
25 it represents in a sense possibly -- "possibly," I say, a

eb35

1 higher distribution or a more single-point type of data than  
2 may be ordinarily available when one cannot have available to  
3 him a single spiral or a single small lot of plate material  
4 made under controlled conditions.

5       What we did was to isolate this spiral of material,  
6 follow it through the manufacturer's process and obtain samples  
7 of the material at various points in the process, including  
8 the samples of the unimpregnated plaque material at various  
9 stages of the impregnation and of the final plate material  
10 prior to formation, final formation and after final formation.

11       I will not make any attempt to show you all the  
12 data that we have but I do want to show you some selected data  
13 which is typical and point out some interesting results that  
14 we are beginning to see.

15       First of all I would like to show you our own ver-  
16 sion of electronmicroscope photographs of two stages of plate  
17 material. One is the unimpregnated plaque and the other is  
18 the finished plate after formation.

19       (Slide-152.)

20       This is a scanning electronmicroscope photograph  
21 of the unimpregnated sinter used to make the positive plate.

22       This is at 100 X as it was printed on the  
23 photograph as you see it there.

24       I might point out that to me this looks like it is

eb36

1 near the same magnification as the photos that Gerry showed  
2 you earlier when he mentioned the figure 500 X. Now I'm not  
3 sure what the difference is there but I believe I'm right when  
4 I say that this one is at 100 X.

5 (Slide 153.)

6 This is the same material, positive plate, un-  
7 impregnated plaque at 1,000 X. Now again here this looks much  
8 larger than the photos that Gerry showed that were mentioned  
9 as 500 X so I'm not sure why there seems to be that difference.

10 But here you can see even more graphically than  
11 you can in the smaller magnifications the type and degree of  
12 variation of void spaces and sizes and shapes and you can  
13 appreciate the problem of trying to evaluate and characterize  
14 the nature of the void spaces in these sintered structures.

15 Next is a photograph, a scanning electron micro-  
16 scope photograph at the same magnification of the completed  
17 positive plate.

18 (Slide 154.)

19 I'm not sure exactly how yet to interpret the photo-  
20 graph on your right, what it all means, but one of the interest-  
21 ing points that I'll point out which I haven't completely  
22 evaluated is the -- are the plateaus that you can see there  
23 where it appears that the high spots have been flattened or  
24 sheared off.

25 This plate material is made by a process which

eb37

1 involves compacting the positive material sinter prior to  
2 impregnation.

3 By the way, the photograph on the left is of a  
4 material that had been compacted and doesn't appear to be --  
5 doesn't have that appearance, so I'm not quite sure yet why  
6 these two figures don't correlate better than they do.

7 The other photograph of that type that I can show  
8 you is a finished negative plate which is roughly similar in  
9 appearance to that of the positive plate.

10 (Slide 155.)

11 There are considerable areas that appear to be  
12 flattened.

13 Now what in general we are trying to do is to --  
14 We're doing two things with the analytical approach to this  
15 plate material. As part of an Air Force-supported program we  
16 are going to do extensive life testing of cells containing  
17 this plate material. This life testing will be under various  
18 conditions and will extend over a period of a minimum of two  
19 years and possibly longer.

20 We expect to be analyzing the condition of the plate  
21 material at intervals during the life test and so the analysis  
22 we're doing now is the reference point to which we are going  
23 to be comparing the additional analyses as time and cycling  
24 progresses.

25 The other thing we're doing is on the internal-

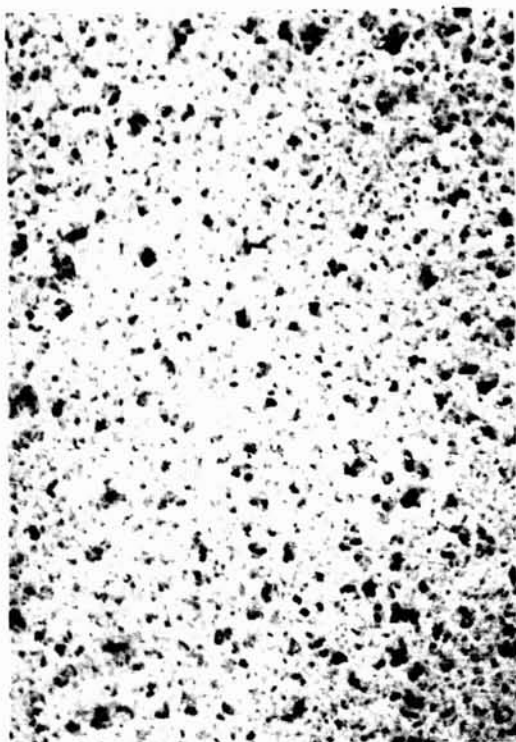


Figure 152



Figure 153

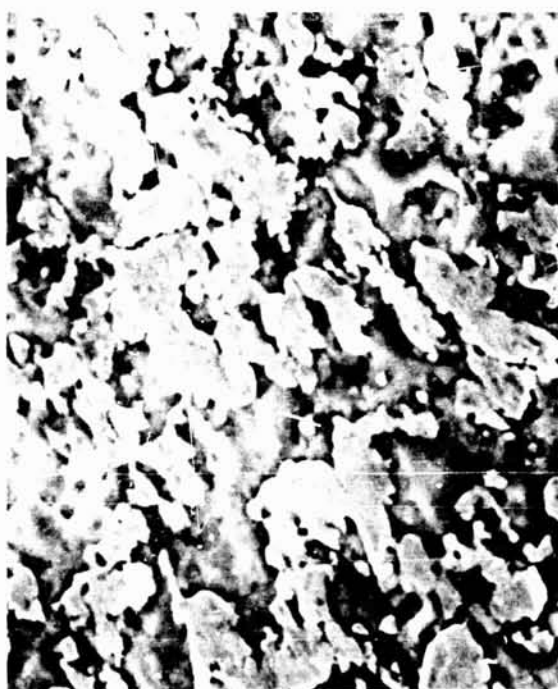


Figure 154

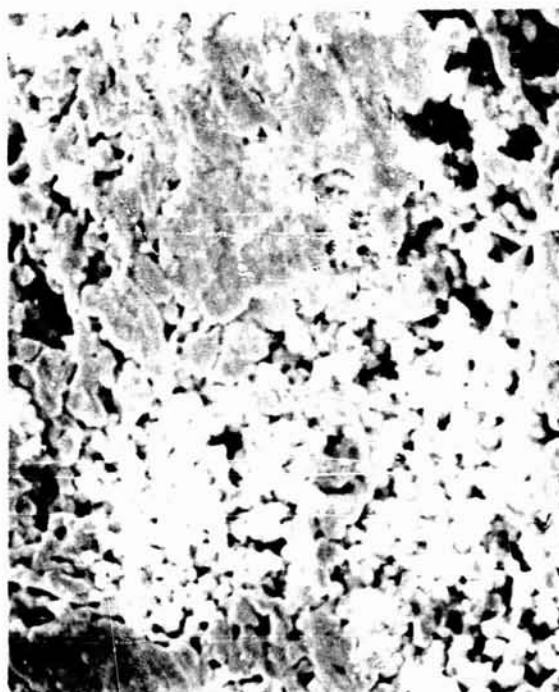


Figure 155

eb38

1 funded program is that we are building some special cells in-  
2 house under laboratory conditions containing this plate  
3 material, among others, and we are going to be cycling those  
4 cells under conditions of 80 percent depth of discharge over  
5 a period of time to determine basically what the mechanism of  
6 degradation is in terms of actual changes in the physical and  
7 chemical conditions of plate materials.

8 We're not restricting ourselves to plate materials;  
9 we are going to be analyzing changes in separator materials  
10 also. So this data is the forerunner of that effort which  
11 will be extending over a period of time.

12 (Slide 156.)

13 I just want to show you a list of the types of  
14 analyses that we are going to run for unimpregnated plaque.  
15 I've put the word "surface" in quotation marks because I am  
16 not quite sure exactly yet what we're going to be able to tell  
17 of the surface by the scanning electron microscope. We're  
18 still investigating that.

19 We are measuring void fraction by absorption of  
20 water. Thickness and weight are of course very simple  
21 characteristics, and purity content involves analysis for  
22 carbon and for various metallic minor constituents by spectro-  
23 graphic analysis.

24 The plate characteristics are essentially those  
25 that most people are thinking about and looking at these days.

eb39

1 I don't think I'll need to comment further on that, but I'd  
2 like to show you quickly if I may -- if the gurgling of  
3 stomachs doesn't drown me out -- one set of data for carbonate  
4 analysis because I would like to make a point here.

5 (Slide.157.)

6 Two points.

7 Now first of all, the two items at the top of this  
8 are for plate material after it is finished with its complete  
9 processing just prior to insertion in a cell.

10 Now notice that the positive plate analysis -- this  
11 is on weight percent -- is higher than the negative analysis.  
12 Now this is interesting because the consensus of a number of  
13 people seems to have been that the problem of carbonate is  
14 always worse on the negatives than it is on the positives and  
15 our data don't seem to bear this out.

16 Now down at the bottom, I have four sets of data  
17 from what I call our negative plate study. This was a set  
18 series of analyses tracking the plate material-- This is nega-  
19 tive plate only in this case. I don't have the data for the  
20 positive yet -- through the process.

21 Now these are slightly out of order at the bottom  
22 but the first line gives the analysis for carbonate prior to  
23 formation. The second line gives you the data for the values  
24 after formation but prior to washing where the plates were  
25 vacuum dried and not contacted with air except for a very few



PLATE CHARACTERISTICS TO BE DETERMINED

UNIMPRGNATED PLAQUE

"SURFACE" BY S.E.M.

VOID FRACTION

THICKNESS

WT./UNIT AREA

IMPURITY CCNTENT

FINISHED PLATES - BEFORE AND AFTER CYCLING

"SURFACE" BY S.E.M.

"SURFACE" AND CROSS-SECT. BY E.M.P.

VOID FRACTION

THICKNESS

WT./UNIT AREA

ACTIVE MATERIAL (CHEMICAL ANALYSIS)

CHARGED POSITIVE ACTIVE MTL.

CHARGED NEGATIVE ACTIVE MTL.

ELECTROCHEMICAL CAPACITY

CARBONATE

NITRATE

IMPURITY CONTENT

Figure 157

CARBONATE ANALYSIS DATA  
(FIGURES IN WT. PERCENT)

IN FINISHED PLATES	CARBONATE ANALYSIS DATA (FIGURES IN WT. PERCENT)	
	AVERAGE*	RANGE*
POSITIVE PLATES	1.12	0.88 - 1.26
NEGATIVE PLATES	0.73	0.64 - 0.78

FROM NEGATIVE PLATE STUDY

BEFORE "FORMATION"	0.80	0.68 - 1.08
AFTER "FORMATION," NO WASH, VAC. DRY	0.89	0.80 - 1.00
AFTER WASH, VAC. DRY	0.35	0.32 - 0.37
AFTER WASH, AIR DRY	0.85	0.71 - 0.96

\* Data from 3 samples selected at random.

Figure 156

eb40

1 minutes required to transfer them to the vacuum drying equip-  
2 ment.

3           The last line should be the next one in order. It's  
4 the analysis for negative plates after completion of the normal  
5 processing including air drying and that figure is really  
6 comparable to the one up at the top. They were plates taken  
7 from a different point in the spiral, and I'm not quite sure  
8 why the range and average is not the same between those two.

9           But that second line and the bottom line are com-  
10 parable points in the process.

11           VOICE:   Excuse me, could you read those numbers?  
12 We can't see them.

13           SCOTT: Yes.   The four numbers at the bottom, the  
14 average is .80, .89, .35, and .85.

15           The second line from the bottom is a special situa-  
16 tion where after normal washing, the plates were vacuum dried  
17 rather than air dried.

18           Now the point is here that it appears that during--  
19 in this particular case, during the process of formation which  
20 involved cycling in concentrated KOH solution, there was  
21 essentially no change. In fact, there appears to be a slight  
22 increase in the carbonate level in these plates.

23           From there to normal air dry, there was essentially  
24 again no change in the carbonate level in the plates. However,  
25 if, instead of air drying, the plates are taken at that point

eb41. 1 and are vacuum dried, there is a significant reduction in  
2 carbonate level down to .35 percent, so this would tend to  
3 indicate that people should give some attention to the exact  
4 method of washing and drying and handling the plates at this  
5 point in time.

6 Just to give you an orientation, the .8 percent  
7 level would give you, if it was all extracted into the electro-  
8 lyte in a normal cell containing a normal amount of electro-  
9 lyte, would give you a carbonate concentration of the order of  
10 90 grams per liter, which I consider to be excessively high.

11 HALPERT: I want to apologize to Will for moving  
12 him a little bit faster than he planned. He had some other data  
13 to show.

14 The cafeteria is expecting us at this particular  
15 time so we'll take a break, and since we have the pre-charge  
16 work to start with this afternoon, I'm sure there is going to  
17 be a great deal of discussion, and I wonder if we could make  
18 it back here by 1:30.

19 (Whereupon, at 12:30 p.m., a luncheon recess  
20 was taken.)

C/9  
ebf 1 AFTERNOON SESSION

2 (1:35 p.m.)

3 HALPERT: If there is anybody who is going to be  
4 going to National Airport after this meeting, we have someone  
5 who is interested in getting a ride. I'd appreciate it if you  
6 would let me know and I'll be glad to pass on the information.

7 For this afternoon's session I'm going to turn  
8 the session over to Dean Maurer, Bell Labs, who will introduce  
9 our speakers and handle the afternoon session.

10 MAURER: We have a special treat for the first  
11 thing this afternoon. We have some movies and Charles Thomas,  
12 Chrysler, is going to show us how to fire up a silver-zinc  
13 battery.

14 THOMAS: This film that we will see documents the  
15 results or partially the results of a program that we are  
16 conducting for the NASA/Marshall Space Flight Center to define  
17 the hazards associated with silver-zinc batteries.

18 The hazards documented by this film are the ones  
19 that are the most spectacular, that is, of spontaneous com-  
20 bustion due to certain abnormal conditions.

21 Now we have had some problems with silver-zinc  
22 batteries in the past and NASA felt that we should evaluate  
23 more thoroughly the conditions that will cause spontaneous  
24 combustion. We have evaluated such things as -- the effect of  
25 such things as insufficient electrolyte, short circuits in

-eb2

1 cells, overloads of batteries, and overheating of cells.

2 The film that you'll see has been partially edited.  
3 It does not include the results of all the tests. It documents,  
4 though, first the results of a short circuit in a 350 ampere  
5 hour cell, then second, the effect of overload on a battery  
6 containing 350 ampere hour cells, and third, the effect of  
7 overload on about a 35 ampere hour battery.

8 In the case of overloads, the actual discharge  
9 current for this test was something like, oh, six times rate  
10 maximum current.

11 In the case of the short circuit in the cell, the  
12 resistance prior to activation of the cell between the positive  
13 and negative terminals was 75 ohms. Now that's one of the  
14 things that was very interesting. The resistance of the short  
15 circuit need not be low in order to cause this problem.

16 Do you want to start the film?

17 (Whereupon, a film was shown with the following  
18 commentary.)

19 The first part of the film shows activation of the  
20 cell. The cell that is under test is in the chamber right under  
21 the clock.

22 The electrolyte was put into a reservoir through  
23 which a vacuum was pulled. The vacuum was then removed after  
24 the air had been evacuated from the cell and the electrolyte  
25 is now being drawn into the cell.

eb3<sup>4</sup> 1 Now the short circuit is directly underneath the  
2 cement that holds the thermocouple on.

3 Now you see the electrolyte beginning to boil in  
4 the vicinity of the short circuit. The temperature -- well,  
5 it's about 150 degrees Fahrenheit.

6 It looks like the editor didn't leave enough of the  
7 temperature gauge.

8 The time to combustion for this cell was about 45  
9 minutes.

10 Now you can see at the bottom light and discolora-  
11 tion, smoke, and it appears from the glow at the bottom that  
12 maybe combustion has actually started on the back side or on the  
13 bottom, although you'll see shortly a bright spot just to the  
14 left of the thermocouple.

15 The temperature at combustion was just about 325  
16 Fahrenheit.

17 Now once these things start burning, combustion is  
18 quite vigorous because of the --

19 (Laughter.)

20 Now this is a single cell. Think of what you'd  
21 have with 21 cells.

22 What happens is that oxygen is liberated by a break-  
23 down of the silver peroxide on the positive plate. This oxygen  
24 supports combustion. Now this is one of the problems which  
25 you have with a silver-zinc battery fire. It's very difficult

-eb4

1 to extinguish, or impossible.

2 This was a pretty good test of that clock, too.

3 (Laughter.)

4 Incidentally, the clock is still running.

5 There's some more of the film. The clock is still  
6 operable. This is the same clock used for battery test,  
7 battery overload test.

8 Now let's see. Watch closely. This is the one that  
9 explodes.

10 Now that was a 35 ampere hour operated at about six  
11 times normal rating. The internal pressures were so high that  
12 a very large contact in the output connector was actually blown  
13 out like a bullet and the lid, the cover, blew off. The cover  
14 has 20 screws and about half of them were broken off; the other  
15 half were just pulled out, the thread stripped off.

16 We've got another test. This is a 350 ampere hour  
17 battery. Now combustion occurred but was not sustained in the  
18 case of this battery because the peroxide had been apparently  
19 virtually all depleted before combustion started.

20 We started discharging this battery at a relatively  
21 low current and did not get up to a current of sufficient  
22 magnitude to cause internal overheating until we had consumed  
23 most of the capacity.

24 Now this battery was being discharged at 350 amps.

25 That is smoke. It's not electrolyte vapor. It had

eb5

1 a distinctive zinc odor that you get from a burning zinc plate.

2 Incidentally, the mode of combustion here appears  
3 to be that the zinc plates start burning first at about 325,  
4 350 degrees Fahrenheit. Of course after they start to burn,  
5 oxygen is liberated and this raises the temperature to the point  
6 where everything inside the battery burns.

7 (End of film.)

8 Any questions?

9 MAURER: Any questions?

10 GROSS: Sid Gross, Boeing.

11 Did you deliberately create a short in the cells  
12 before you --

13 THOMAS: Right.

14 GROSS: -- filled with electrolyte?

15 How did you do that?

16 THOMAS: We drilled a hole through the side of the  
17 plastic case between two of the plates, and of course the hole  
18 went down into the area between the plates. We then inserted a  
19 stainless steel pin that fit rather loosely. We wanted to have  
20 a fairly high resistance short.

21 GROSS: A stainless steel pin between all the plates?

22 THOMAS: No, between just two of the plates.

23 The short circuit-- It appears that you have a  
24 greater tendency for combustion when the short circuit area is  
25 small.



e56

1 GROSS: Had you ever seen or heard of any similar  
2 fires occurring naturally?

3 THOMAS: Yes. Afterwards you might correct my  
4 memory on this but I know of at least three cases in which  
5 batteries of the type used in the Saturn launch vehicles have  
6 burned spontaneously due to problems that we were never really  
7 sure of.

8 MAURER: Any other questions?

9 We have a slight reorganization-- We have another  
10 question for you.

11 PASCHAL: I might add a little bit to this.  
12 On the batteries that had failed previously we had an estimated  
13 unit battery that had been a spare unit for one of the launches.  
14 After the launch, this battery was brought back up to Marshall  
15 and was put under vibration test. It was then pulled to a  
16 laboratory and was sitting in the laboratory, and of course  
17 the shelf life of this battery wet is about 120 hours, so  
18 obviously the shelf life had long been exceeded.

19 As a result, the cell failed through failure of  
20 the separator system. This was one of them that Charlie Thomas  
21 is talking about.

22 A second battery was an S-4-B stage battery which  
23 failed during test operations on the West Coast. This particu-  
24 lar one failed as a result of a controller, heat controller  
25 that stuck and caused the heater to fail to turn off. This

ea7 1 heated the battery up-- There's a heater blanket in the  
2 bottom of this battery and of course with the controller turned  
3 on, the heater remained on until it was able to heat the  
4 battery to such a temperature as to cause the battery to catch  
5 fire.

6 MAURER: We had a reorganization of the program  
7 slightly. This afternoon was originally scheduled for thermal  
8 properties and so we'll actually adhere to that.

9 We have one or two papers on thermal properties.  
10 That will get rid of all the fires and get the smoke out of  
11 the room. It'll get the heat off and then we can get down to  
12 business and have the rest of the day and into the night to  
13 haggle over pre-charge effects.

14 So the next speaker will be Bill Webster on the  
15 design and development of the heat flow calorimeter for  
16 measuring thermal properties of aerospace cells.

17 WEBSTER: The co-author of this paper is a fellow  
18 by the name of Sid Teller, also a Goddard employee, and any  
19 questions that you might have, if you cannot reach me, I'm sure  
20 you could also get ahold of Sid. His number is 5379.

21 The development of the calorimeter was decided  
22 because people are unable to predict from theory the amount of  
23 heat that will be generated by a nickel-cadmium battery. This  
24 is extremely important to the spacecraft manager and to the  
25 battery engineer since, as we know, a battery's operational

eb8

1 life is proportional to its temperature.

2 For instance, at 15 degrees C. we might expect a  
3 five-year life out of a cell. At 35 degrees C., this life  
4 would diminish to one year.

5 For a period of over 60 years, scientists have been  
6 building coulometers, so why did we build another one? Well,  
7 we thought that of all these coulometers that appear in  
8 the literature, about 400 of them, none of them were particu-  
9 larly suited to an aerospace cell.

10 The problem is further complicated by the fact that  
11 in the next decade, NASA will be using at least seven different  
12 sized cells in their spacecraft ranging from a 6 ampere hour  
13 to a 100 ampere hour sized cell, so what we wanted more than  
14 just a coulometer, which we've had before -- I did some work  
15 at American University on one; Joe Cherfee has done some work  
16 on it, and quite a few other people -- is we wanted a univer-  
17 sal coulometer, one instrument bought one time that could  
18 handle all size cells, and that's what we got.

19 We had a six-month contract with Rocketdyne which  
20 called for the calibration of the instrument at Goddard, the  
21 design and construction of the instrument.

22 I would like in my first slide to show you a com-  
23 parison between a 6 and a 100 ampere hour cell, and discuss  
24 the design problems confronting an instrument maker.

(Slide 158.)

ebg 1 In case some of you have forgotten, a 6 ampere hour  
2 cell only weighs 250 grams, and the 100 weighs almost 4,000  
3 grams, so the first problem that you have is one of geometry,  
4 thermal mass, and response.

5 For instance, we might anticipate that the 6 ampere  
6 hour cell would rate one watt where the 100 ampere hour cell  
7 -- we didn't really know but we speculated as high as 25 watts.  
8 Also, as you know, you have to bring leads into and out of the  
9 cell. This could cause two sources of error in the coulometer.  
10 One would be  $I^2R$  heating and the other would be conduction  
11 heating.

12 Again as you are aware, cells operate in a con-  
13 straint manner so that if a designer were to operate a cell  
14 on a coulometer in the same manner in which he would operate  
15 one in a spacecraft or on your lab bench, you would have some  
16 kind of massive constraining plates, especially on that 100  
17 ampere hour cell.

18 This would be extremely bad insofar as it would  
19 slow down the thermal response of the cell.

20 Also, we wanted this coulometer to be capable of  
21 operating at minus 10 degrees Centigrade through to plus 40  
22 degrees C., have an accuracy and reproduceability of 1 percent,  
23 and a response time of less than five minutes.

24 All these criteria were met and in my next slide  
25 I'd like to show you the philosophy of design that met this.

ebf0

(Slides 159, 160.)

This is just a schematic and the detailed drawing of the coulometer appears on that chart over there. It is so involved that I think the schematic is all that we can show.

First of all, we have an adiabatic shield which isolates the instrument from the environment. This, through differential thermocouples, maintains a temperature equilibrium of approximately 5/100ths of a degree Centigrade.

Then we go internal to the calorimetry vessel itself. Now this is where the first design criteria was met.

This is a pressure vessel. We filled the vessel with helium. Helium has excellent thermal properties and eliminated the need for constraining points on the cell.

How would the coulometer operate? All right, if you would like to look at a cell, you would place it on a silver platform in the coulometer and if you'd like to investigate it at, say, 25 degrees C., that would be T-2 in this equation on the left.

Then coming down from the silver tray you have a pair of copper rods. They go down to a heat sink. This heat sink can contain many materials but for the purpose of this discussion we used liquid nitrogen. That's  $T_1$ . That is a fixed temperature.

The next thing that we have is K which is the conductivity of the copper rod, A, the cross-sectional area,

Feb 11 1 and X, the length.

2 Now the first thing that is done is that you go  
3 through a calculation here that says you need so many watts.  
4 You apply the wattage. In this case it would be 30 watts to  
5 the automatic heater at the top. There is a thermister circuit  
6 there which senses the temperature and keeps the bridge in  
7 balance.

8 Now once equilibrium is established at that tempera-  
9 ture you initiate your cell reaction and the amount of heat  
10 that is liberated or absorbed by the cell during this reaction  
11 is detected by this thermister bridge and that quantity of heat  
12 is either added or subtracted to the automatic heater, and this  
13 is a direct measure of the rate of reaction that takes place  
14 in the cell.

15 Now I'd like to point out some of the unique features  
16 of this design.

17 This is actually two copper rods separated by a  
18 thin mylar film. They serve as your positive and negative  
19 terminals. They are sized so that you have no  $I^2R$  heating  
20 even at 100 -- well, at 50 amps was the requirement.

21 Also, the heat conduction is working for you. It is  
22 one direction. It is from the coulometer to your heat sink.

23 Additionally you'd say, well, what happens if you  
24 want to look at a 6 ampere hour cell on this same coulometer  
25 instead of a 100? You would simply use a different eutectic

6512 1 material here. You'd use something like ice water.

2 Ice water would then give you  $T_1$  here. The amount  
3 of wattage that you'd need to maintain that equilibrium condi-  
4 tion at 25 degrees C. would be, instead of 30 watts,-- I  
5 haven't gone through the calculation but it would be consider-  
6 ably less, like 5 watts.

7 Also, something I would not recommend but another  
8 thing you could do, you could adjust the sensitivity by adjust-  
9 ing the length of the rod.

10 There are a lot more details on the picture, on  
11 the poster over there which are omitted from this drawing.  
12 For instance, there is a pressure vessel which goes around the  
13 outside which has cooling coils so that your adiabatic shield  
14 is always applying heat to isolate the system from the environ-  
15 ment.

16 There are other things to take care of radiative  
17 heat losses. The pressure vessel is completely gold-plated.  
18 In addition, the copper rod coming down is protected by several  
19 radiation shields plus a vacuum chamber so that you don't have  
20 heat losses there.

21 We have been using this coulometer here at Goddard  
22 in support of the Houston program with Grumman. We've been  
23 looking at the 100 ampere hour cell.

24 One of the first things we did was Grumman fur-  
25 nished us with a heater cell which was made by Eagle Picher

eblj

1 Corporation which-- It removed one positive and two negative  
2 plates and substituted an expanded nickel material in there.

3 We used this cell to measure the response time of  
4 the calorimeter.

5 Slide off, please.

6 (Slide 161.)

7 And this is what we found.

8 Now there is a calibration heater in the calorimeter  
9 located at the base of that silver tray. This was used to  
10 calibrate the instrument without a cell in there and we found  
11 that it met the design criteria of less than 5 minutes.

12 Now this is what happens when you add a large  
13 thermal mass, like a 100 ampere hour cell. You can see that  
14 it takes approximately 90 minutes for 90 percent of your signal  
15 to come out. Now this was done at 25 degrees C., and a con-  
16 stant 20 watts was put in the coulometer. And then 100 percent  
17 of your response requires 140 minutes.

18 Next slide, please.

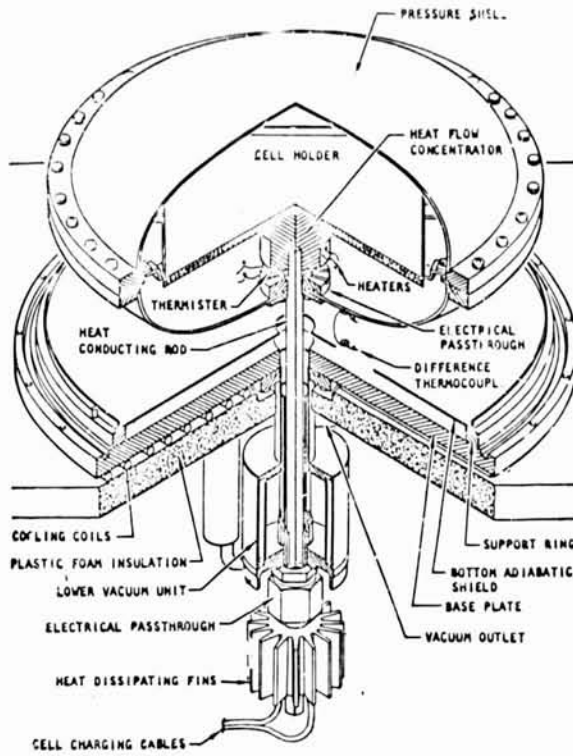
19 (Slide 162.)

20 The next thing we did is we took a General Electric  
21 cell in this case, a 100 ampere hour cell, and we subjected it  
22 to a near-earth orbit, a 90-minute orbit, 110 percent recharge  
23 and 25 degrees C. What we were setting was the effect of  
24 thermal output versus depth of discharge. The percent recharge  
25 was fixed; the temperature was fixed. It was 10 percent, 15





Figure 158



Quarter Section Drawing of Calorimeter

Figure 159

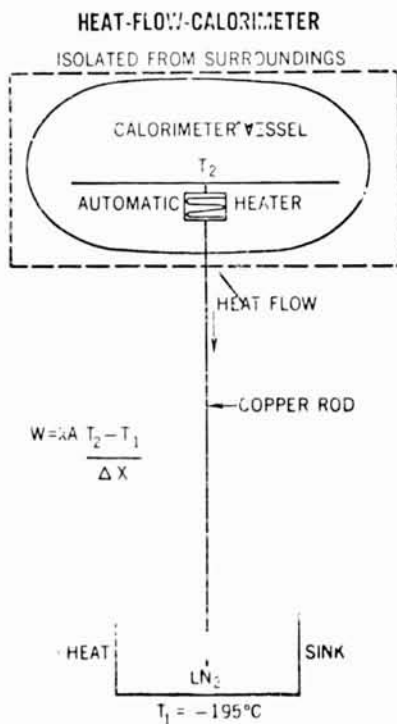


Figure 160

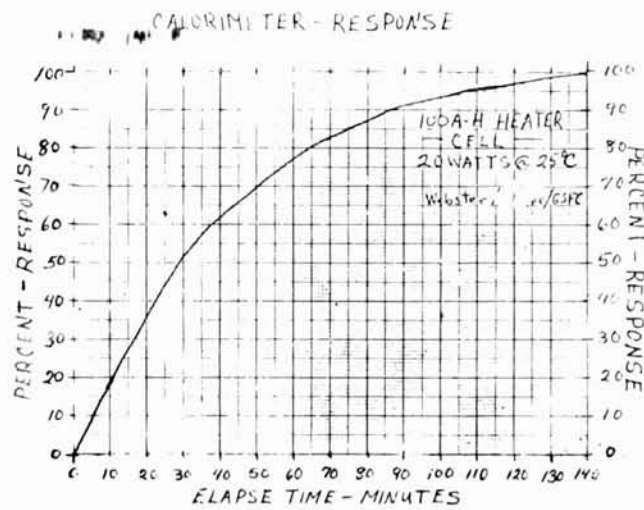


Figure 161

eb14

1 percent. 20 percent, 25, 35, and 45 percent.

2 Now remember, this is a 30-minute orbit so that  
3 45 percent, we're talking about 90 amps.

4 You will also notice that the curves are changing  
5 shape. This seems to be following a trend so that you could  
6 predict possibly what was going to happen. Then at the higher  
7 current rate something else happened.

8 We ran a check of the instrument to make sure it  
9 wasn't instrument error by using a copper bar across the output  
10 terminals and applying the same amount of current and there was  
11 no error associated with that amount of current.

12 My next slide.

13 (Slide 1637)

14 What I did was I plotted what I call inefficiency  
15 where I took the peak thermal output versus the integrated  
16 watt hours during discharge and you can see what is happening.  
17 At a discharge rate of 20 amps with 10 percent depth of dis-  
18 charge, we're talking about an inefficiency which people talk  
19 about on OAO, about 15 percent.

20 But as your rate goes up, your inefficiency goes  
21 up and when we get up to 90 ampere hours, we're up as high as  
22 32 percent. Now this actually I would think is almost common  
23 sense. It's a current density problem that we're probably  
24 running into here.

Another consideration, though, and that may be more

15 1 important to a thermal engineer, would be what is the average  
2 bay temperature, so I integrated the total heat out during that  
3 30-minute discharge, put that over the electrical wattage,  
4 and we end up with a completely different picture. We're  
5 talking about a range from about 6 percent to a maximum of 10  
6 percent.

7 Not being a thermal engineer, I don't know which  
8 one of these figures would be more significant and I don't know  
9 in the past, when people referred to the thermal inefficiency  
10 was typically 15 percent, which figure they were referring to,  
11 but it's a distinction you have to make. If you're interested  
12 in peak output, it's considerably different. It's as much as  
13 25 percent higher.

14 Next slide, please.

15 (Slide 164.)

16 Then of course everyone always says, "Well, what  
17 happens during overcharge," and we thought we'd take one more  
18 look at it, and it happens just the way everyone always says  
19 it does.

20 What we have here is we have a continuous overcharge  
21 at various rates, C over 10, C over 20, C over 40 and C over  
22 80, and we find in each case that once the cell has become  
23 fully charged and you've hit an equilibrium condition, your  
24 thermal energy and your electrical -- your thermal and your  
25 electrical are exactly the same; that is, you multiply your

eb16<sup>1</sup>

1 current rate times your cell voltage and you're right on.

2 The little discrepancy here is probably due to the  
3 fact that when we get down in this range we should have changed  
4 the sensitivity of the coulometer. We were up at the 30 watt  
5 baseline; we come down here; we're looking for less than two  
6 watts out of 30.

7 Next slide.

8 (Slide 165.)

9 This is the last slide. This is looking at the  
10 thermal output as a function of the percent recharge. Here  
11 we kept the depth of discharge constant. We kept the tempera-  
12 ture constant and we put in different amounts of energy, 105,  
13 110, 115 percent. And you can see that there is definitely an  
14 increase also with the percent recharge, but the order of  
15 magnitude is much less than that that I had in the first slide  
16 where we were talking about the effect of the depth of dis-  
17 charge, so it is a factor.

18 Of course, if we continue to overcharge the cell  
19 then it would become a very predominant factor but we are not  
20 in this particular experiment; only up to 115 percent.

21 We have right now-- We've been running this instru-  
22 ment on a continual basis since January. We have about 125  
23 pages here of raw data that we're reducing and we intend to  
24 come out with our next report but to be honest, it will probably  
25 be spring before it comes out, so if anyone is interested in

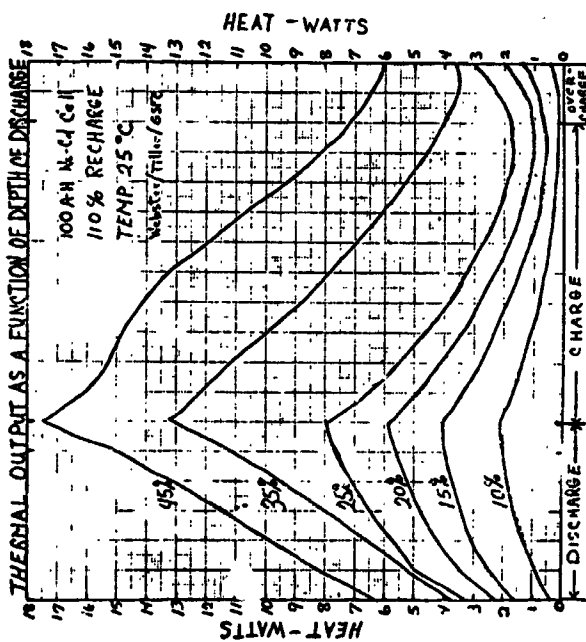


Figure 162

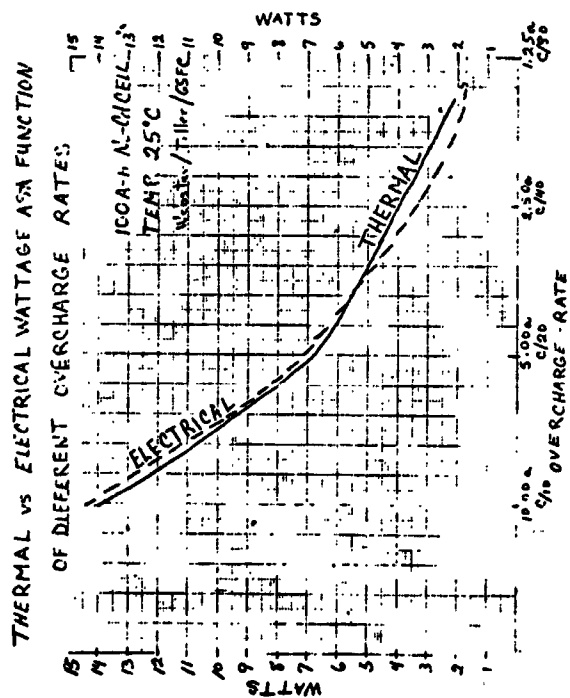


Figure 164

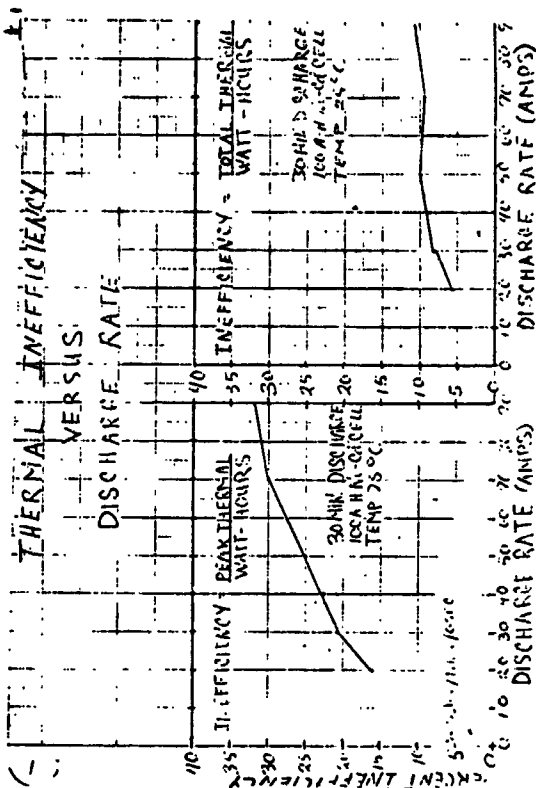


Figure 163

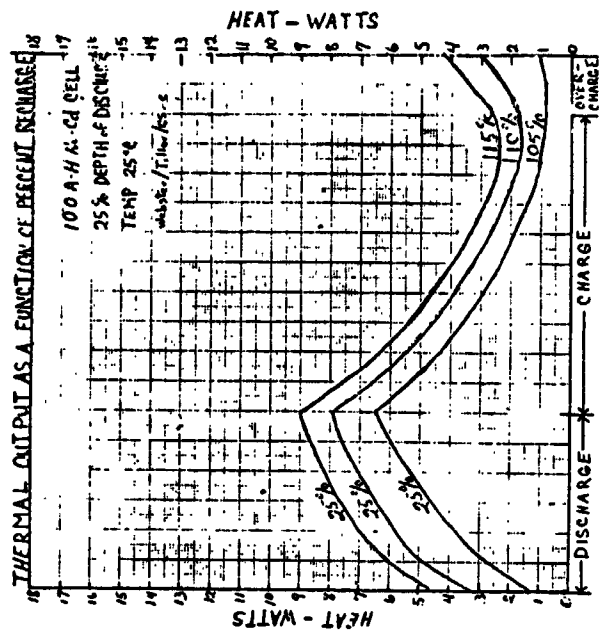


Figure 165

eb17

1 obtaining more information, we have it here and we just haven't  
2 had time to get it published yet.

3 MAURER: I have a question to start off, Bill.

4 In some of the curves that you showed it would ap-  
5 pear that the time constant of this system was something on the  
6 order of an hour and yet you were showing heat flow data that  
7 was considerably less than an hour, like 30 minutes. How do  
8 you know whether what you're measuring is controlled by the  
9 system or whether it is controlled by the cell?

10 WEBSTER: Your observation is correct. We cannot  
11 do anything about improving the response of the system since  
12 the thermal mass of the cell governs that. However, the system  
13 is an equilibrium system. It operates at 25 degrees C., and  
14 on the C over 2 discharge to one volt where we are going for  
15 a period of two hours, the curve of our heat output follows  
16 exactly the curve that we obtain from a heater cell.

17 The only thing I could say regarding this, and it's  
18 probably not really answering your question, is that if at the  
19 end of 30 minutes you were to go back to your calibration curve  
20 and you'd see this represents some percentage of the total out-  
21 put, maybe 60 percent, if you were to continue at this rate to  
22 one volt which would take maybe two hours or longer, you would  
23 see that these two values concur.

24 So I don't think the instrument is influencing it  
25 and it is a valid criticism, though, that you're not seeing all

ab18 1 the heat, but it is due to the mass of the cell.

2 GROSS: Sid Gross, Boeing.

3 I've calculated the thermal response of cells, not  
4 that one but others, quite large, approaching that, and it's on  
5 the order of four or five minutes so therefore, the one hour  
6 time response of your instrument appears to be mostly due to  
7 the design of the instrument and not the response of the cell.

8 It is possible to closely approximate the thermal  
9 response of the cell with other design approaches.

10 WEBSTER: Sid, a question on that. What order of  
11 wattage are you talking about and how did you run your ex-  
12 periment?

13 GROSS: Thermal response is calculable and it's  
14 not a function of the heat rate. It's the time required to  
15 approach one over pi on the final asymptot of the result.

16 WEBSTER: And you think a cell of this size has a  
17 thermal response of less than five minutes?

18 GROSS: Well, I haven't calculated that but you  
19 could easily calculate it. It's quite straightforward.

20 WEBSTER: What size cell are you talking about?

21 GROSS: I've calculated it on a cell about an inch  
22 and a quarter thick.

23 WEBSTER: That's the same thickness. But what about  
24 the rest of the geometry?

25 GROSS: Well, I think this dimension turns out to be

eb19 1 the most important.

2 WEBSTER: We're talking about 4,000 grams of weight  
3 here.

4 GROSS: Well, the thermal response is a function  
5 of the thickness squared and the thermal diffusivity and the  
6 conductivity, and you can do it fairly straightforward by  
7 calculation and get a pretty close estimate of what the response  
8 of the cell is.

9 Or I guess you can actually run an experiment where  
10 you separate the cell from the coulometer in an experiment  
11 designed specifically to measure the thermal response of the  
12 cell --

13 WEBSTER: That's right.

14 GROSS: -- and that would confirm or get a more  
15 accurate value. And I think if you do this that you will be  
16 able to identify most of your one hour time response as a  
17 built-in design feature of the coulometer.

18 WEBSTER: I will certainly look into this. I think  
19 it has been looked into. I will have to look back into the  
20 reports. The fellow who did this is Dr. Bill Johnson, who is  
21 internationally known for his work in coulometry.

22 He did go through such a calculation. I believe it  
23 appeared in the first report concerning the design of the  
24 instrument, and we have operated it without the cell in there  
25 and the response time has been less than several minutes, and



eb20 - 141  
✓  
1 with the cell in there then the response time has gone up to  
2 the times indicated. And the servo loop for the coulometer  
3 has a response time on the order of seconds, so I don't really  
4 think --

5 GROSS: The calculations I've made on this kind of  
6 thing, the heat transfer between the cell surface and the  
7 heat sink is the critical step.

8 MAURER: Bob?

9 STEINHAUER: Steinhauer, Hughes.

10 Bill, your data is 25 percent at the discharge and  
11 I assume you are concentrating on that for obvious reasons,  
12 but will you be generating or are you in the process of  
13 generating data for deeper depths, namely geared at synchronous?

14 WEBSTER: Yes, in the near future here we're going  
15 to be supporting the ATS and OSO project here at Goddard, and  
16 the ATS is a synchronous orbit program. We'll be looking at  
17 15 ampere hour cells, 50 percent depth of discharge, and  
18 various other depths of discharge, and supporting also the SMS  
19 program here at Goddard.

20 I might also mention that this instrument is avail-  
21 able for uses by private industry insofar as requesting us  
22 to run some cells for you in it. And sometimes we might not  
23 be able to accommodate this by schedule and we might be able  
24 to take a cell which we have in there, like for instance the  
25 15 for ATS, and approximate your regime while we have that

ehz1 1 cell in there.

2 MAURER: Our next speaker is Joseph Yuen who will  
3 talk about controlled battery temperature in satellites with  
4 Berlon and Berlox.

5 YUEN: We all have heard about batteries. Since  
6 we are the space satellite battery user, I'm going to give you  
7 some information and results from the battery that is now on  
8 the satellite in orbit.

9 Most of our battery packs are 918 F size poly-  
10 propylene triple-sealed cells. The size of the pack is  
11 roughly 5-1/2 inches by 7-1/2 inches in diameter and weighs  
12 about 15 -- 11-1/2 pounds.

13 All cells are supported by an echo form mode and  
14 in the early part of our program we put this mode with the  
15 battery pack inside of a sealed container. In the last two  
16 launches we have witnessed a rise in battery temperature and  
17 this is the configuration of our battery pack.

18 (Slide 166.)

19 Here is the battery pack, the F cells inside with  
20 the nylon insulator and this whole pack is inside a sealed  
21 container. And we monitored the battery pack with a tempera-  
22 ture sensor that was buried inside of the echo form. At that  
23 time we did not monitor the temperature on the cell itself.

24 So a year ago our battery pack witnessed a high  
25 temperature on the payload. We were not able to command the

eb22 1 TM on so we're guessing the temperature is a little bit high.

2       So on our last launch, solar F-10 which launched  
3 from Wallops Island in July of this year, four months ago,  
4 we feel that the battery problem-- The battery was kind of  
5 hot so we used another method of trying to conduct the heat  
6 away from the battery pack.

7       We are also monitoring the temperature on the cells,  
8 on top of the cells. On the battery pack we have two rings  
9 of cells, an inner ring and an outer ring. We also monitored  
10 the cells, each cell on the inner ring and the outer ring.

11       The battery pack is also supported the same way  
12 with echo form but we add an aluminum plate underneath with  
13 the nylon separators. But we also did away with the cover --  
14 I mean the hermetic seal cover because we feel that the cells  
15 themselves are hermetically sealed so why launch the battery  
16 pack with a sealed cover?

17       So instead of insulating it with the nylon we used  
18 a Berlon -- Berlox disk, which is a ceramic disk, beryllium  
19 oxide filled ceramic, and the glue the ceramic onto the bottom  
20 of the cell and also onto an aluminum substrate. We glued it  
21 with beryllium-filled oxide RTV Berlon. They call it Berlon.  
22 These two items are made by National Beryllium Oxide up in  
23 Haskell, New Jersey.

24       We've got temperature from the payload now, so  
25 it's a very comfortable temperature.

eb25

1 (Slide 167.)

2 At 62 percent sun the battery pack ranged from 21  
3 to 24 degrees C.; at 63, from 25 to 28; at 90 percent sun it's  
4 34 to 36. And I just got word from the office a while ago  
5 that this payload now is in 100 percent sun and the battery  
6 has only gone up as high as 38 degrees C.

7 The rest of the payload is pretty cool.

8 I think the battery pack sits on a support tube  
9 that mainly supports the rest of the electronics in the pay-  
10 load and the support tube is also mounted to a thick plate of  
11 honeycomb, and that's what the electronics are on. And the  
12 honeycomb plate is only seeing about 21 or 26 degrees C. And  
13 this method we used to carry heat away shows very good results.

14 Now I would also like to mention something about  
15 the rectangular cells. This was on the round cells.

16 On the rectangular cells I have now worked out a  
17 method using annodized shim to go between each cell. As we  
18 all know, hard coat annodized aluminum is a good insulator but  
19 it is also a good thermal carrier. So in between cells we have  
20 a thin film of annodized shim and we clamp them this way. We  
21 have to clamp them anyway so we might as well clamp them and  
22 carry heat down to another heat sink plate and it will be  
23 mounted into our honeycomb substrate by that method to carry  
24 heat away.

25 (Slide 168,169.)

eb24

1 On the side of the cell we will also clamp it to  
2 another annodized shim to carry heat away further. We feel  
3 that this method of controlling the temperature in the battery  
4 in the payload has been quite successful. This is the method  
5 we have hoped to carry on.

6 That's all.

7 Any questions?

8 GROSS: Sid Gross, Boeing.

9 I didn't understand how you get the heat out from  
10 the bent shim to the bottom. Is it bonded on?

11 YUEN: The bent shim is this one right here,  
12 actually, and this is another-- You have another heat plate  
13 which is right underneath the bent shim.

14 GROSS: The two are bonded then?

15 YUEN: This is also bolted to another big plate  
16 that is also a support plate of the payload.

17 GROSS: This shim then is bolted to something under-  
18 neath it?

19 YUEN: Right.

20 GROSS: On the outside, on the outside flange?

21 YUEN: No, no. These flange are the bolting flange  
22 and also hold the cell down to the plate.

23 GROSS: Okay.

24 One comment on the use of annodizing aluminum --  
25 annodized aluminum for an insulator. This is kind of an iffy

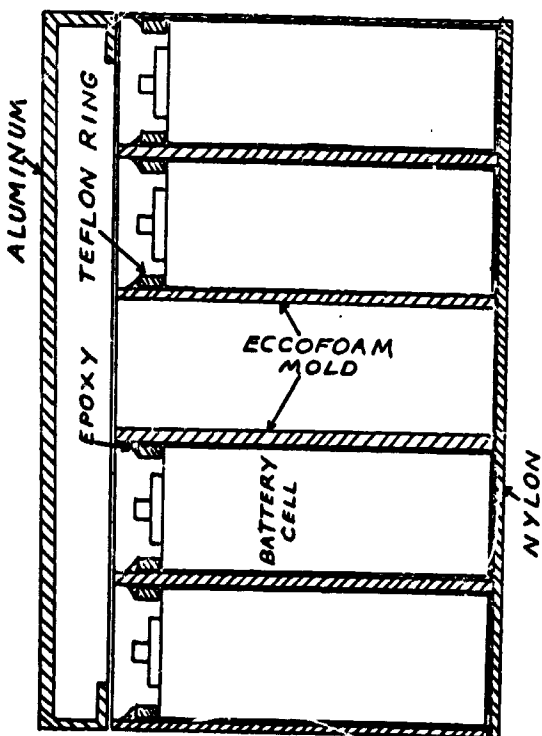


Figure 166

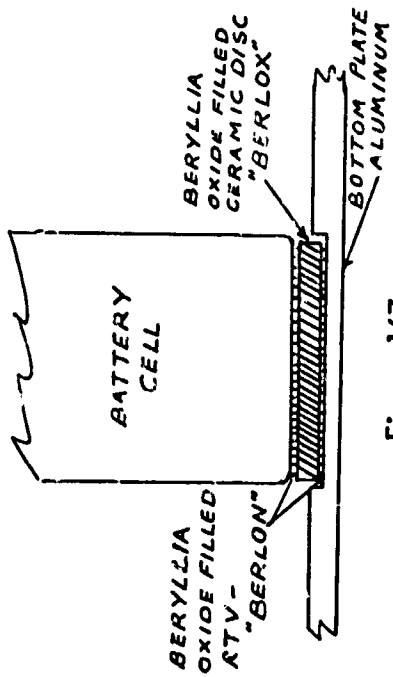


Figure 167

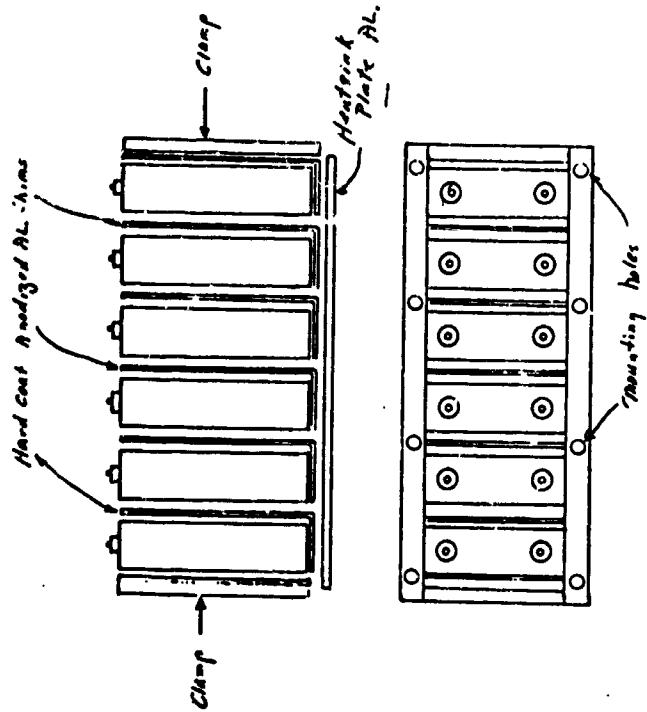


Figure 168

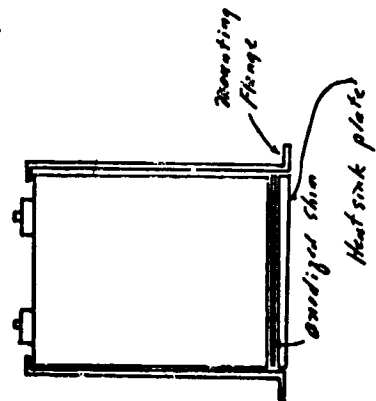


Figure 169

eb25

1 thing sometimes and it might warrant looking into. It is hard  
2 to depend on consistent protection at battery voltages.

3 YUEN: We plan to use a two-terminal cell and so  
4 I figure the voltage between the case and the inside won't  
5 be too great.

6 GROSS: As I recall, the anodizing films break  
7 down around 15 volts.

8 YUEN: But I'm quite sure you'll have about maybe  
9 a tenth of a volt in between.

10 GROSS: I guess it would be worth looking into.  
11 Sometimes you can get a whole battery voltage across the  
12 insulator in certain failure modes.

13 MAURER: The highest plate voltage in the cell will be  
14 sitting between the cell case, inside the cell case and the  
15 case in contact through this aluminized plate to the ground?

16 YUEN: Yes, but we also have insulation inside the  
17 cell.

18 MAURER: But that's in contact with the electro-  
19 lyte so-- insulation inside the cell can will be in contact  
20 with electrolyte and that can only stand 1.2 volts per cell  
21 until you get hydrogen.

22 CARR: Earl Carr, Eagle Picher.

23 It seems to me that probably the spacecraft is  
24 grounded so that whenever you have a series string of cells  
25 you have built up a potential and you've got voltage to that

Feb 26 1 case through the electrolyte path and you can have a breakdown  
2 of electrolyte and that is a single point failure.

3 YUEN: We have measured hard core annodized. Hard  
4 core annodized they claim is roughly about 3 mils thick and  
5 I used an ohmmeter with two scribes in my hand and I couldn't  
6 even break through to read any kind of a resistance.

7 STROUP: Gene Stroup from Goddard.

8 Joe, it seems to me I recall reading some reports  
9 from TRW Systems regarding an attempt to annodize on the OGO  
10 battery, I believe it was.

11 Would anybody from TRW like to address the subject  
12 of your annodizing on that particular project?

13 I don't know what the annodizing-- I don't know  
14 how you did that or what the thickness of it was. That might  
15 clarify this point that Joe has brought up.

16 SCOTT: Scott from TRW.

17 Since you've put me on the spot, Gene, I have to  
18 say something, --

19 (Laughter.)

20 -- and that something is that I wasn't around when that was  
21 going on.

22 (Laughter.)

23 But I'll say one other thing and that is that I  
24 concur with the people who are concerned with the high voltage  
25 available at the positive end of a multicell series string



eb27 - 1  
1 battery and also there is another potential problem and that  
2 is during ground handling or if you have any -- especially if  
3 you have any electrolyte leakage -- perish the thought -- you  
4 can get high conductivity through the pores of some of these  
5 annodized films. That will be an accelerated point of break-  
6 down of the annodizing and you can really have problems.

7 But I don't remember anything-- I don't have any  
8 knowledge of what the story was on the OGO program, Gene.  
9 Sorry.

10 STROUP: One other thing on that. TRW did use  
11 a fiberglass sort of insulating material between the annodized  
12 structure and the cell, the annodized heat transport fin and  
13 the cell. It seems to me-- I did not measure the depth of  
14 the annodizing itself but it seems to me that it was thinner,  
15 much thinner than what you described, Joe, and it might be  
16 worth looking into.

17 I believe I may have some of these in the lab.

18 YUEN: Yes, but when you're using a fiberglass  
19 insulating you're defeating the purpose of that annodized  
20 can carrying thermal conductivity.

21 STROUP: Yes, but I understand it was necessary in  
22 this particular case.

23 MAURER: One more question before we go on to the  
24 next, and coffee after that.

25 GASTON: Gaston, Grumman.

eb28

1 I have a comment. On the OAO batteries we have  
2 certain similarities to your packaging and certain differences.  
3 We do have a hard coat annodized heat fin, L-shaped, but before  
4 we take the cell we wrap it in a layer of Kapton, about one  
5 mil Kapton to insulate it. Then we embed it in a Dow Corning pot-  
6 ting material to smooth the surface of the cell so we get a  
7 good thermal contact.

8 So we do have more than one insulation. We do  
9 have the hard coat that's an insulation and we do have the  
10 Kapton and additionally, we have the Dow Corning potting.

11 And at the bottom of the L shape that is mounted  
12 directly against a heat sink, again we have an insulation there.  
13 I think it is also Dow Corning, where it is mounted against  
14 the heat sink.

15 So you have various numbers of insulation and  
16 additional electrical leakage path protection.

17 YUEN: One more thing I forgot to mention.

18 (Slide 169.)

19 Besides the annodized we were also anticipating  
20 a surface that would be not flat so we were also going to  
21 use some of these in between so that will probably still further  
22 carry the heat away and take care of any voids that there  
23 is.

24 MAURER: One more question from Steinhauer.

25 STEINHAUER: Steinhauer, Hughes.

eb29

1 Without telling tales out of school, I do recommend  
2 that you look carefully into insulating of cells electrically.

3 One other possibility is if a cell case does get  
4 grounded, what is the potential of that case with respect to  
5 the electrodes in that cell and what type of gases could be  
6 generated.

7 MAURER: The next presentation will be by Dan  
8 Lehrfeld, whom Steve Gaston had mentioned would tell us about  
9 the thermal design of the Grumman 100 ampere hour battery.

10 LEHRFELD: I have requested some time in this  
11 session to provide some additional background information on  
12 a subject that Steve Gaston first introduced yesterday, and  
13 that is the work we've been doing at Grumman on the design  
14 of a 4-cell module for a manned orbital space station.

15 I'll present some of this additional material and  
16 then perhaps if there are any questions, even left over from  
17 yesterday or that may arise today, I'll be happy to answer  
18 them.

19 Basically the goal of our work at Grumman is to  
20 design a 4-cell module which can be a building block to a  
21 space power system for a 25-kilowatt manned orbital space  
22 station. I have some notes here that perhaps I could project  
23 on the screen. It might help familiarize some of you with  
24 the manned space station program if you are not already so  
25 familiar.

eb30

(Slide 170.)

The space station program being studied for NASA by McDonnell Douglas and North American-Rockwell considers a manned system with an average power level of 25 kilowatts. What this means to the battery system and to backtrack, the power system for this station is foreseen as a solar cell nickel-cadmium battery system.

A typical battery system would consist of 148 of our 4-cell modules or 672 cells. This corresponds to an average 30 percent depth of discharge in orbit.

To give you some feel for the size of the system, that number of 100 ampere hour cells weighs approximately three tons and we're talking about a total module weight of on the order of four tons, allowing about 35 percent for packaging.

The battery voltages in the system and the actual system configuration varies with the study. The different contractors are looking at many different systems. Typically we have seen systems described as 21-module batteries, eight such batteries comprising the total system, or 28-module batteries, these modules strung in series, six such batteries comprising that particular system.

So you can see that we're talking about a fairly large number of cells and the battery system does represent a significant part of the over-all space station program.

(Slide 171.)

BACKGROUND INFORMATION ON  
MANNED SPACE STATION PROGRAM

POWER LEVEL: 25 KW (average)  
POWER SYSTEM: Solar array/Ni-Cd battery  
BATTERY SYSTEM: 168 -4 cell modules (672 100 AH cells)  
(based on 30% average DOD, 94 min. orbit)  
BATTERY WEIGHTS: 672 cells (@ 8.5 lbs./cell) 3 tons  
168 modules (@ 33% packaging wgt.) 4 tons

BATTERY SYSTEM

CONFIGURATIONS: Vary with electrical system concept  
TYPICAL CONCEPTS: a) 8-21 module batteries in parallel  
b) 6-28 module batteries in parallel

Figure 170

100 A.H. MODULE DESIGN REQUIREMENTS

MECHANICAL: a) 0 psia to 100 psig internal cell pressures  
b) Saturn type vehicle launch loads

ELECTRICAL: a) 4-100 AH Ni-Cd cells connected in series  
b) 12 to 50% orbital DOD  
c) Case to case (and case to ground) electrical isolation

MAINTAINABILITY  
& HUMAN FACTORS: a) Module removal (and installation) without breaking  
coolant lines  
b) Target Weight: 45 pounds

THERMAL: a) Heat removal via a circulating water cooling loop  
b) 32 to 55°F inlet water temperature @ 30 lb/hr. flow  
rate  
c) 0 to 14.7 psia compartment pressures  
d) 0 to 1g gravitational field  
e) Allowable cell (case) temperature range 32 to 68°F  
(0 to 20°C)  
f) Minimize cell to cell temperature gradients

Figure 171

eb31

1 This next page will outline some of the requirements  
2 that have been defined for us in the design of this module.

3 Mechanically we've designed it for internal cell  
4 pressures ranging from zero psia up to 100 psig and addi-  
5 tionally we've designed for the type of launch loads you would  
6 expect from a Saturn-type vehicle.

7 Electrically we have four 100 ampere hour sized  
8 cells connected in series. Our orbital regime varies from  
9 12 to 50 percent depth of discharge over a 94-minute orbit.

10 Additionally, we must provide case-to-case isola-  
11 tion and case-to-ground isolation within the module.

12 We also have maintainability and human factors  
13 considerations because each of these modules is replaceable in  
14 orbit. The entire battery system over the life of the space  
15 station system will probably be replaced many times. That is  
16 the reason for packaging this four cells as the basic unit.

17 Two requirements which we thought of putting down  
18 here just off the top of our heads were the target weight in  
19 our program which is approximately 45 pounds, and that's come  
20 out of human factor studies which have been done which have  
21 shown that that's approximately the maximum weight that an  
22 astronaut in orbit should be required to handle.

23 Additionally, we just design our module to mount  
24 with a cooling system and to be removable without breaking any  
25 fluid lines in that system.

eb32 1           Some of the thermal requirements that we have to  
2 contend with are we are designing for heat removal via a  
3 circulating water system. There is a water system aboard the  
4 space station in the baseline studies through which all elec-  
5 trical equipment dissipates its excess heat.

6           We are designing for a range of inlet coolant  
7 temperatures from 32 up to 55 degrees F. at a flow rate of 30  
8 pounds per hour available per module.

9           Additional requirements are that the modules be  
10 mounted in crew-habitable compartments which can be up to  
11 14.7 psia environment or during a depressurized operation could  
12 be zero. And the gravitational field in the space station can  
13 be either zero G or up to one G.

14           The requirements on our cells are that the package  
15 must maintain cell temperatures no higher than 68 degrees F.  
16 or 20 degrees C. while the package is exposed to the maximum  
17 environment. Our minimum temperature is 32 degrees F. although  
18 in the space station that requirement is automatically met.

19           What I have down there is cell case temperature.  
20 When we speak of cell temperature we refer to the temperature  
21 on the cell case; that's our reference temperature.

22           Additionally, because we have four cells packaged  
23 and we have a moving coolant system picking up the heat that  
24 we're rejecting, there will be some variation in temperature  
25 from cell to cell, and we've tried to minimize that variation

eb33

1 so that the four cells are as nearly isothermal as possible.

2 (Slide 89.)

3 I have a Vu-graph which we'll put on now that shows  
4 the 100 ampere hour module that we've constructed for engineer-  
5 ing evaluation tests. The picture was taken during fabrica-  
6 tion; it's not complete but I think it can illustrate the  
7 design to you.

8 We call this the flat-pack design. I should back-  
9 track to say that in our studies we have considered many dif-  
10 ferent geometric arrangements and we've looked at alternate  
11 possibilities in packaging the module. However, the thermal  
12 requirements are pretty stiff; that is, we've got to maintain  
13 the maximum cell case temperature at 68 degrees with a coolant  
14 inlet temperature of 55, so we've only got a total thermal  
15 gradient of 13 degrees allowable.

16 So some of the more traditional packaging methods  
17 that we've looked at such as packing cells broad side to broad  
18 side with thermal conduction fins in between and channeling  
19 heat down to a base plate -- we've looked at those but those  
20 resulted in very large, massive thermal conduction plates be-  
21 cause of the size of the cell.

22 We're talking about a large amount of heat and the  
23 height of the cell being approximately 7-1/2 inches high, that  
24 heat has a long distance to travel to a typical base plate  
25 mounting system. That would result in a very large gradient



eb34 1 over the face of the cell and to counteract that you would  
2 require very massive plates.

3           The design that we've come up with here is lighter  
4 in weight. It meets the requirements. It comes very close to  
5 our goals, and we think it's a very simple package because it  
6 is easy to fabricate; it doesn't require internal heat pipes  
7 which is one of the things we did consider in our program but  
8 the fact that we do not require them we think is a plus for  
9 this design.

10           To describe it for you, we have the four cells end-  
11 to-end, resulting in a very long, narrow package. The heat  
12 dissipated within the cells primarily comes out the broad faces  
13 of the cells across an interface which consists of Kapton  
14 tape and capsule material, a hard anodized coating, to the  
15 side walls of the module.

16           It is conducted down the side walls to the mounting  
17 flange. This is the mounting flange, one on each side of the  
18 package. The heat is channeled to this mounting flange. It's  
19 rejected through a bolted interface to a cold rail.

20           This cold rail typically would be a long aluminum  
21 extrusion which provides a passage for a flow of coolant past  
22 the module and it also provides structural support for the  
23 module in a typical system.

24           The cannister itself is made out of 6061 T-6  
25 aluminum. This side plate you see here, 30 inches by

eb35

1 approximately 8 inches, is one integral piece of aluminum  
2 that has been machined out. These are stiffening ribs required  
3 to constrain the cell pressures that we're designing for.

4 The module mounts to this cold rail via captive  
5 hardware which is not shown in this drawing, and this is early  
6 in its fabrication. The captive hardware consists of bolts  
7 which would be installed here. They move with the module and  
8 they simplify the ease of installation of the module in that  
9 the module just has to be slipped in between the two cold rails  
10 and a normal Phillip's head type screwdriver used to fasten  
11 the module to the cold rails.

12 We use a silicon grease interface material between  
13 the cold rail and module to enhance the film performance of  
14 the interface.

15 What's shown-- Structurally, let me say this:

16 We provide-- Our basic cannister provides for  
17 containment of the cell on five sides. This top cover which is  
18 installed last is bolted down and constrains the sixth side, so  
19 we do constrain all six sides in our design.

20 It is hard to see in this picture but each of the  
21 cells is in an individual compartment. There is a base plate.  
22 There are end plates, and there are cross pieces separating  
23 these into four separate compartments.

24 What is shown here are tabs which are welded to the  
25 side plates. They structurally connect these cross pieces to

eb36

1 the side plate making it a one-piece, rigid construction.

2 To speak just about the fabrication techniques,  
3 the way the module is built up is after assembly of this basic  
4 cannister, we would take a premeasured amount of potting  
5 material. We did study various potting materials. We selected  
6 one which has very good heat transfer properties. It has a K  
7 of 0.5 btu per hour per foot, and very low viscosity which  
8 was important, as I'll get into later.

9 We would measure out predetermined volumes of this  
10 potting material. After it is catalyzed we would pour it into  
11 the bottom of each one of these cannisters. The cell would  
12 then be taken-- The cell is potted with an internal vacuum  
13 in the cell. It would be taken and placed down into the cannis-  
14 ter to sit on top of that layer. Then it would be allowed to  
15 sink under its own weight, allowing the potting to rise around  
16 all four edges of the cell.

17 Then we apply pressure to the top of the cell until  
18 it is fully seated. By that time the potting will have flowed  
19 up all around the cell and we guarantee that we are encapsu-  
20 lating the entire cell.

21 The final step after that potting hardens, we would  
22 top pot and then install the cover.

23 Let me just mention, the cells that are shown here  
24 are the heater cells which were constructed for us by Eagle  
25 Picher. That's these two extra set of terminals you see here.

eb37

1 Those are the terminals by which we energize that heater cell.

2 (Slide 172.)

3 This Vu-graph just summarizes the status of our  
4 program, where we've actually gone to.

5 We've designed, fabricated. We've assembled an  
6 engineering test module with four actual cells. We've selected  
7 our configuration which is the flat pack; it has been optimized.  
8 We do feel it is the optimal consideration based on weight,  
9 thermal performance, maintainability, and the structural re-  
10 quirements.

11 We have run an engineering evaluation program of  
12 both thermal tests to demonstrate the design the proof pressure  
13 tests. These have been completed and the module has performed  
14 within our expectations.

15 MAURER: Thank you.

16 I think in order to keep within our time frame --  
17 A few people have flights to catch earlier -- or later, rather,  
18 in the afternoon -- let's not have questions now. In fact,  
19 let's have coffee --

20 (Laughter.)

21 -- and perhaps the thermal discussion can go on in small  
22 groups.

23 (Recess.)

24

D ebl<sup>u</sup>

1 MAURER: We've finally made it to the star attrac-  
2 tion of the session, the pre-charge session. And the first  
3 speaker will be Dr. Font from SAFT, who will speak on a study  
4 of different pre-charge levels in 20 ampere hour cells.

XZXZX

5 FONT: I will try to present to you some results on  
6 the pre-charge study performed on the 20 ampere hour  
7 cells under a contract program or contract study.

8 In the preliminary study we have determined that  
9 after formation cycles on the plates, cadmium remains on the  
10 plates and this has been determined by chemical analysis to  
11 be from four to seven ampere hours on our 20 ampere hour cells.

12 I have to state that we have negative to positive  
13 ratios in these cells, theoretical ratio, theoretical capaci-  
14 ties of 2.0 which is real capacities are about 1.7.

15 We have performed three different levels of pre-  
16 charge in three different groups in these cells and we have  
17 performed this pre-charge using a venting technique. I will  
18 show you the results.

19 (Slide. 173.)

20 On oxygen evolution on all these cells of the con-  
21 tract, we see in this table that the rate of oxygen evolution  
22 is about twice between all the cells from 300 cubic centimeters  
23 to 600 cubic centimeters.

24 (Slide. 174.)

This table represents results on pre-charge

eb2

1 measurement. We have made in all the study two different  
2 measurements, one by chemical analysis and one by electro-  
3 chemical measurements.

4           You see on the first table there the results on  
5 ampere hours of the cells -- ampere hour determination of cad-  
6 mium, inactive cadmium on the plate. We have put on these  
7 cells, E group, four ampere hours corresponding to oxygen;  
8 ten in F group, and 16 in group G.

9           So before doing anything else, in each group we  
10 have total pre-charge of 1 to 11, 14 to 17, and 20 to 23. In  
11 these cells we have performed a cycling test, 30 cycles and  
12 each cycle's duration is 12 hours, 11 hours charge at zero-10  
13 and one hour pre-charge at zero-2 rate.

14           (Slide 175.)

15           Here are the pressure evolutions expressed in abso-  
16 lute pressure versus the time. And we see that in the group  
17 of higher pre-charge, the pressures are lower than they are  
18 in the other pre-charge adjustment. This is on cycle 2.

19           (Slide 176.)

20           And we have another sheet where we see on cycle  
21 29 it is almost the same. We have to notice a little dif-  
22 ference in the end of charge pressure, more sensitive in G  
23 group than in any other; very little change.

24

25

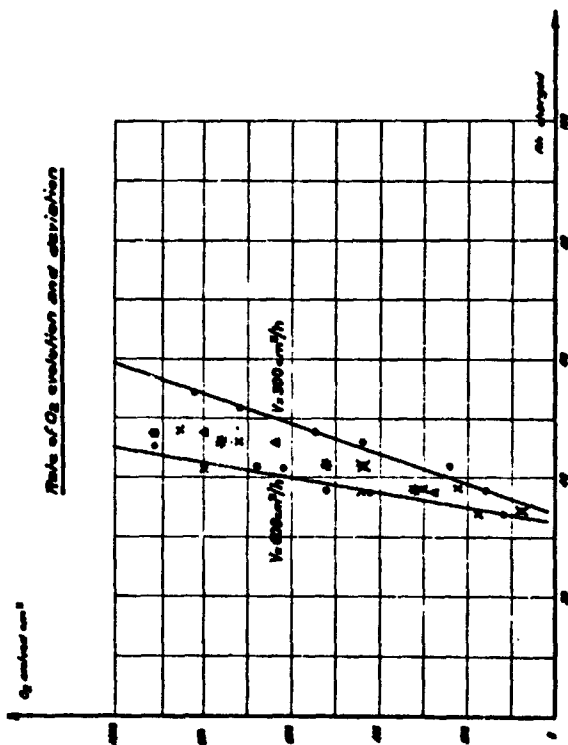


Figure 173

PERFORMANCE ANALYSIS

	E	F	G
<u>Pressure evolution</u>			
Chemical analysis after formation	4-7	4-7	4-7
Precharge by venting technique	4	10	10
TOTAL PRECHARGE	8-11	14-17	20-23
<u>Measured precharge before cycling</u>			
Electrochemical	1.8	4.5	10.4
Chemical analysis	11.3	11.0	9.3
TOTAL PRECHARGE	13.1	15.5	19.7
<u>Measured precharge after 20 cycles</u>			
Electrochemical	0.6	14.2	20.0
Chemical analysis		in process	

Figure 174

Pressure evolution on cycle #12

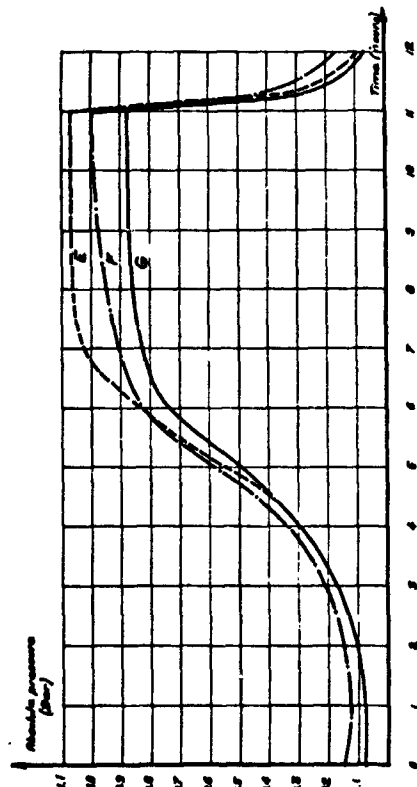


Figure 175

Pressure evolution on cycle #122

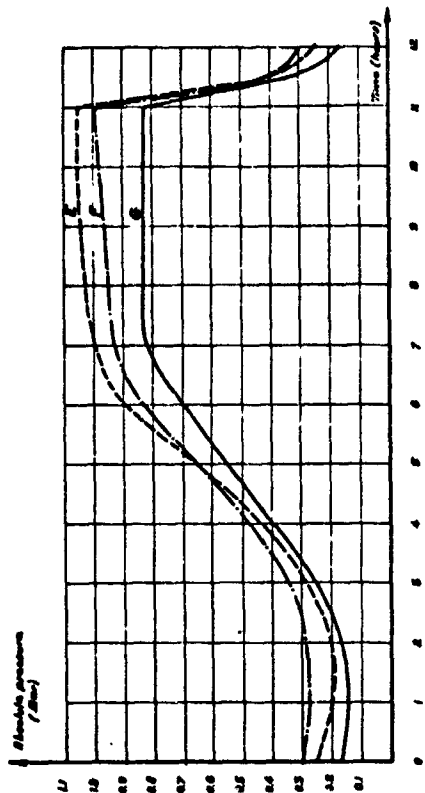


Figure 176

eb3

1 following curve.

2 (Slide 174.)

3 We'll see this later, another time.

4 I will present the first result here.

5 Before doing these cycles, we have measured the  
6 ampere hour electrochemical measurement on each group, and we  
7 have found the following results which are less than the ampere  
8 hours we have put at the beginning on these cells.

9 We have performed chemical analysis on the same  
10 cells and found the following results. This is that electro-  
11 chemical measurements show it's less than what we have put on  
12 the cells but that chemical -- cadmium has increased. But we  
13 have to note that the total amount of pre-charge is about the  
14 same as we had before, except perhaps on this group which is  
15 a little higher, but we have to take into account the accuracy  
16 of measurements, both electrochemical or chemical.

17 Now we are on the step of measuring electrochemical  
18 and chemical pre-charge in these cells after 30 cycles and we  
19 see that electrochemical has increased in each of these groups  
20 from 1.8 to 8.6, 4.8, 14, 10.4, 20. We have not yet performed  
21 the chemical analysis on these cells. The test is running.

22 The important point is that you have an increase  
23 in electrochemical measurement on each group and this increase  
24 can be explained in the following ways.

25 (Slide 177.)



eb4

1 First of all, we can explain it by the same explana-  
2 tion that Dean Maurer said this morning about the activation  
3 of chemically determined inactive cadmium.

4 The second point, cadmium oxidation of separator.

5 And the third point, nickel attack on positive  
6 electrode by oxygen.

7 So we have not performed the chemical analysis on  
8 the cells after the cycling. We are not able to say exactly  
9 what are one of the many possibilities here.

10 But I have to go back to the discharge curve after  
11 cycling in which we see that the capacities on the high pre-  
12 charge group is higher to one volt -- is higher for the high  
13 pre-charge level and they follow on the pre-charge measurement.

14 (Slide 178.)

15 So seeing this curve, I should say several points.

16 First of all, our pre-charge level, considering  
17 the end of charge pressure and capacity after cycling tests  
18 seems to be good for the cell -- yes, for the cell.

19 A lower pre-charge level gives lower capacity and  
20 higher pressure but if we consider the increase in pre-charge  
21 measurement, electrochemical pre-charge measurement, we see  
22 that high pre-charge levels are very dangerous for the cell  
23 because we need either for long-lifetime cycling overcharge  
24 protection which is discharged cadmium.

25 And as we have not all the results on the chemical

POSSIBLE MECHANISMS FOR INCREASING  
ELECTROCHEMICAL PRECHARGE

1. ACTIVATION OF CHEMICALLY DETERMINED INACTIVE CADMIUM.
2. OXIDATION OF SEPARATOR.
3. NICKEL ATTACK ON POSITIVE ELECTRODE BY OXYGEN.

Figure 177

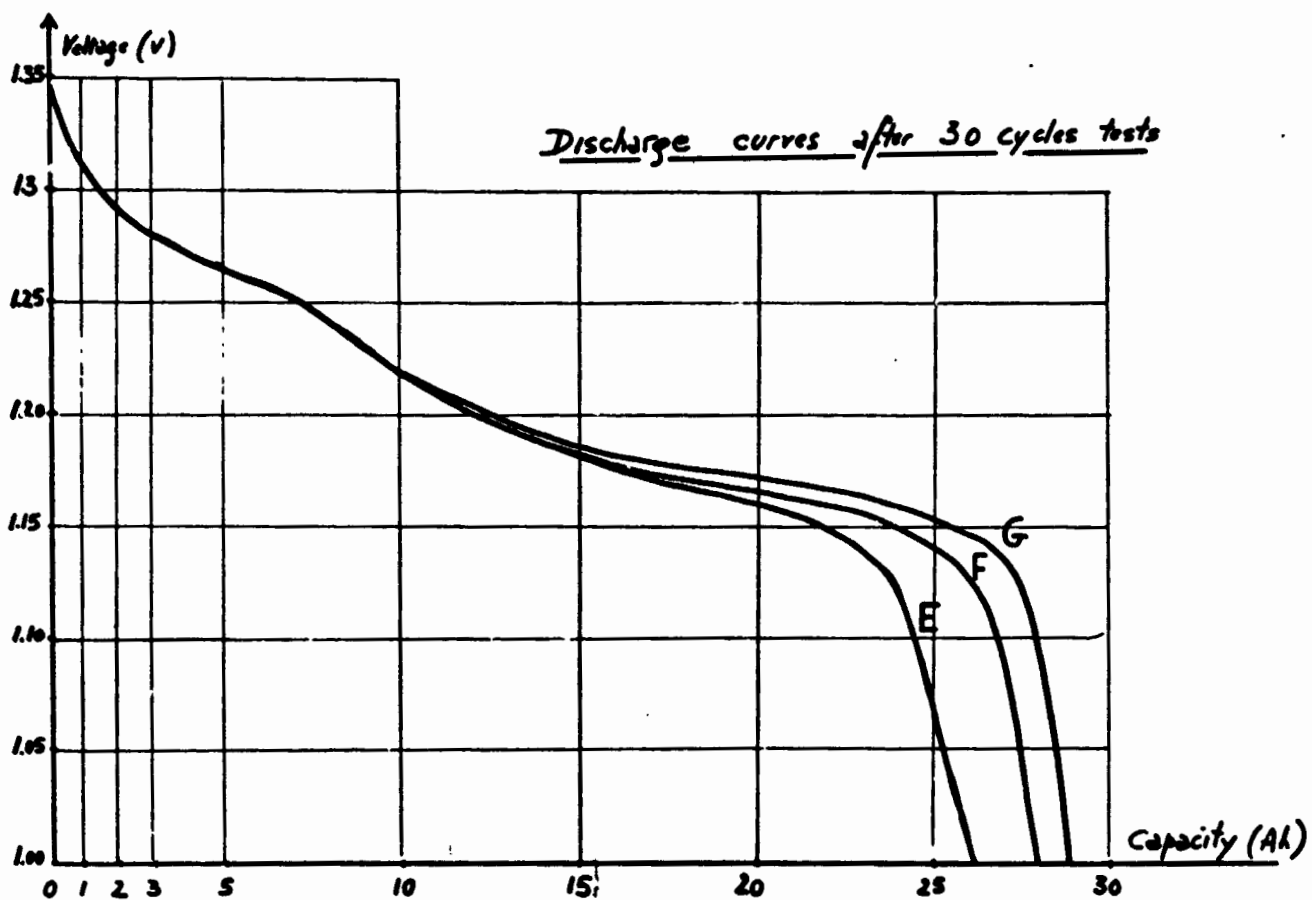


Figure 178

eb5

1 analysis from these cells, we cannot make a further statement  
2 or hypothesis on this point.

3 Thank you.

4 MAURER: Exercising the privilege of office, I will  
5 ask the first question.

6 (Laughter.)

7 How did you measure the electrochemical capacity  
8 of the negatives in the cells?

9 FONT: We measured the electrochemical capacity--  
10 We drilled a hole on the cell after discharging it to one volt  
11 and we put the cell in open condition in potassium hydroxide  
12 and we reversed it versus the mercuric reference electrode and  
13 we measured the capacity to minus 0.5 volts

14 O'ROURKE: O'Rourke, Grumman.

15 I have two questions I want to ask you.

16 You said on the one chart you had up there that  
17 you had put a certain amount of pre-charge in the cell. Did  
18 you determine this by a rate/time method or did you measure the  
19 amount of gas coming out of the cell or --

20 FONT: Putting on the pre-charge, we measure the  
21 amount of oxygen. In the slide I have shown only the beginning  
22 until 1,000 cubic centimeters which is the same extrapolation  
23 for the highest pre-charge level. It's about 3,000 cubic  
24 centimeters at pre-charge.

25 O'ROURKE: How many cubic centimeters of oxygen per

eb6

1 ampere hour? What was the relationship you used?

2 FONT: We used one gram of oxygen, one gram of  
3 oxygen, 3.3 ampere hours.

4 O'ROURKE: Okay.

5 The other question I had was what type of time  
6 period elapsed from the time that these plates or cells were  
7 immersed in electrolyte from the time that you made the actual  
8 electrical pre-charge measurement? Was there any attempt to  
9 control that time?

10 FONT: After formation cycles? After formation  
11 cycling through the electrolyte?

12 O'ROURKE: Did you take a pre-charge measurement  
13 directly after the pre-charge adjustment or did they go through  
14 formation cycles first?

15 FONT: After pre-charge adjustment we closed the  
16 cell and we performed two cycles at C over 10. The pre-charge  
17 measurement was made immediately after the first pre-charge,  
18 after pre-charge. Immediately after pre-charge using venting  
19 technique, we closed the cell. We performed a decharge to one  
20 volt and at this time we determined the electrochemical capacity.

21 O'ROURKE: Okay.

22 The reason I bring this up is I'm going to present  
23 something in a minute. I've found the correlation between the  
24 soaking time, in other words, the time you actually fill the  
25 cell with electrolyte, to the time that you do the pre-charge

eb7

1 adjustment. I've found out there is a relationship between the  
2 amount of pre-charge a cell will have, and that's why I was  
3 just wondering whether that time was either controlled or  
4 measured.

5 FONT: Going into details, after which -- after  
6 formation cycles we assemble the cell and we fill with electro-  
7 lyte. Between performing any charging cycle we keep them 72  
8 hours in that state after we perform one preliminary cycle in  
9 a closed condition and after we perform the pre-charge adjust-  
10 ment.

11 CARR: Ear. Carr, Eagle Picher.

12 I couldn't see all the numbers but just simply:  
13 Were the plates all the same before you set pre-  
14 charge? In other words, was there the same amount of cadmium  
15 in all of the cells?

16 FONT: In all this contract the plates were of the  
17 same lot.

18 CARR: Okay. Very good.

19 And then the next thing is what was the voltage  
20 difference during the overcharge for the three levels of pre-  
21 charge?

22 FONT: We didn't perform overcharge, a long over-  
23 charge time in these cells but the end of charge --

24 CARR: Well, what I was referring to is you  
25 presented some pressure data and corresponding to that pressure

eb8

1 data, what was the difference in voltage between the cell that  
2 was pre-charged?

3 FONT: I think it was not higher than 10 milli-  
4 volts, 10 to 20 millivolts between the different groups. We  
5 had to state that the end of charge pressure are no very  
6 different, too. There is only 200 millibars.

7 KIPP: Ed Kipp, Gulton.

8 Can we assume these are starved cells?

9 FONT: Yes.

10 DUNLOP: Jim Dunlop, Comsat.

11 One point, partially in answer to the gentleman's  
12 question in front here, that Dr. Font and I had talked about  
13 before his presentation. I think I'm going to add something  
14 I think he meant to say.

15 These pre-charge measurements that he is talking  
16 about were made -- the measurements were made after the pre-  
17 charge was set. He got different results-- If you'll notice  
18 the first time he set the pre-charge he put in four -- in one  
19 case he put in four ampere hours of pre-charge and only  
20 measured 1.8 electrochemically.

21 In answer to your question, he also did a chemical  
22 analysis and he found that the remaining -- he found that it  
23 was essential for him to determine the total amount of pre-  
24 charge that he had in that cell at any time to do both the  
25 chemical and the electrochemical analysis. And by doing just

eb9

1 the electrochemical measurement he could not determine how much  
2 pre-charge he had in those cells at any time.

3 STEINHAUER: Steinhauer, Hughes.

4 Dr. Font, could you tell us what method of chemical  
5 analysis was used? Was it complex symmetric titration or  
6 calometric?

7 FONT: It was titration by EDTA method.

8 SEIGER: Seiger of Heliotek.

9 Could you tell us whether you have the same amount  
10 of electrolyte in each of those groups before you set the pre-  
11 charge?

12 FONT: Before we set the pre-charge we have exactly  
13 the same amount of electrolyte.

14 BETZ: Betz, from Fairchild.

15 The initial chemical pre-charge that you showed  
16 there for the seven ampere hours, that's the valuability from  
17 the end of your formation process?

18 FONT: This is the difference on all-- We have  
19 performed these measurements on about 20 plates and this is  
20 the total deviation of the measurements.

21 BETZ: Coming out of formation?

22 FONT: Immediately after formation.

23 BETZ: Okay.

24 The second question: After your discharge to one  
25 volt, do you resistively load the cell to zero volts before you

ob10 1 measure your pre-charge? That is the Goddard technique --

2 FONT: At what stage?

3 BETZ: After discharge to one volt, the technique  
4 for pre-charge measurement has included an intermediate step  
5 of resistively loading the cell until it goes to zero volts.

6 FONT: After discharging to one volt, we have dis-  
7 charged at C over 10 rate from one volt to zero.

8 BETZ: Then C over 10 from one volt to zero.

9 FONT: And after, from zero to minus 0.5 C over  
10 10, too.

11 BETZ: C over 10 also. Thank you.

12 LACKNER: Lackner, Canadian Defense Research.

13 You mentioned you didn't have too much of an over-  
14 charge period; is that correct?

15 FONT: Yes-- Well, not performed especially an  
16 overcharge measurement on these cells.

17 LACKNER: Our experience has been that if we had  
18 too much of a pre-charge that we would get into hydrogen  
19 generation and the way we tested it out was to put the cells on  
20 120 hour overcharge at minus 5, and if you had any hydrogen  
21 generation it most certainly would come out.

22 We feel that you have to tie in the pre-charge with  
23 a long overcharge as a screening out test.

24 FONT: It's not understood exactly --

25 LACKNER: We find that there is a definite



cbl

1 dependence of pre-charge and overcharge capability and parti-  
2 cularly if you are getting into synchronous orbit where you may  
3 have to be charging for long periods of time. I'm not saying  
4 you have to, but you may have to.

5 FONT: You may, yes.

6 LACKNER: We would like to have a cell that could  
7 stand the overcharge capability for a long length of time.

8 FONT: Yes. In this case you have not to put too  
9 much pre-charge.

10 LACKNER: In most of our past experience with a  
11 bare minimum pre-charge you get very good results by stringing  
12 out with a long overcharge period.

13 FONT: Yes.

14 One point here is that I think for synchronous  
15 orbit or a long-life cycling program we have to consider  
16 immediately on the discharge curve that if we have too low pre-  
17 charge the end of charge will be lower in that case. If we  
18 see that curve, --

19 (Slide 178.)

20 -- this curve is only for 30 cycles, and we see that mostly  
21 if you cut here, you have less capacity with lowest pre-  
22 charge.

23 So I think we have to have some compromise between  
24 no pre-charge and high level pre-charge which-- Perhaps for  
25 commercial cells high level pre-charge is good because you have

eb12-2

1 good capacity but no time duration guarantee.

2 But in this case we have to do some compromise and  
3 I think Mr. Dunlop will tell you about this following this  
4 program to determine what amount would be necessary.

5 HALPERT: Halpert, from Goddard.

6 Were these your aerospace grade or aerospace  
7 quality materials that were used?

8 FONT: We used our space quality plates which all  
9 were high ratio-- We used exactly the same materials, nickel,  
10 but we have the space definition with high ratio on these cells.

11 HALPERT: And what kind of separator was it? Nylon  
12 or polypropylene?

13 FONT: Until now we used nylon but we are studying  
14 the eventual change to polypropylene.

15 BELOVE: Belove, Marathon.

16 What is the difference in capacity of those cells  
17 with the low pre-charge and the cells with the higher amount of  
18 pre-charge to one volt?

19 FONT: There is on this curve -- Immediately after  
20 discharge the difference is about two or three amp hours.

21  
22 You have your 26 to 29, about.

23 BELOVE: Thank you.

24 KIPP: Ed Kipp, Gulton.

25 Have you been able to generate enough data so that

eb13

1 you could establish a relationship between the amount of pre-  
2 charge measured electrochemically and that measured by chemical  
3 analysis?

4 FONT: What kind of-- For example, I think an  
5 important point would be the chemical measurement after cycling  
6 tests.

7 KIPP: What I'm getting at is could you-- If you  
8 could establish a definite relationship by that measured  
9 electrochemically and that measured by chemical analysis, you  
10 wouldn't have to do the second, or the first.

11 FONT: Yes, or maybe. You have to point out here  
12 that these two total measurements are pretty close and then  
13 the order of accuracy, but we have to consider this measurement  
14 also, and the possibility of extra pre-charge such as the  
15 nickel attack of the positive electrode by oxygen evolved and  
16 oxydation of the separator with life.

17 So you cannot only consider-- For instance, you  
18 cannot only consider electrochemical pre-charge measurement.  
19 You have to perform the chemical analysis.

20 RAMPEL: Guy Rampel, General Electric.

21 I would like to ask Joe Lackner this question:

22 You mentioned that you have a screening overcharge  
23 at minus 5 for 120 hours. At what rate is that, Joe?

24 LACKNER: C over 10.

25 RAMPEL: Thank you.

eb14

1 FORD: Ford, NASA/Goddard.

2 To what do you attribute the inability or the lack  
3 of being able to measure that pre-charge in an electrochemical  
4 or in your reverse mode?

5 FONT: Really, I don't know. For the moment we  
6 don't know enough to attribute something. Perhaps-- I don't  
7 know.

8 FORD: I'm sure there are other people here who have  
9 experienced the same results. I'd like to throw the question  
10 to the floor.

11 BETZ: This is Betz at Fairchild, and I'll sit for  
12 this one.

13 I'm going to make some general comments. We at  
14 Fairchild are buying cells right now and are experiencing  
15 a very similar type of data. Our test technique for pre-  
16 charge, as I indicated previously, is slightly different so  
17 that the electrochemical measurements of pre-charge we'd be  
18 getting would be less than yours by the difference between  
19 perhaps one volt and zero volts. The one-ohm technique of  
20 getting down there takes out I think a reasonably large amount.

21 We had used initially two ampere hour oxygen pre-  
22 charge and got zero measurable electrochemical pre-charge.  
23 In addition to that, the cell capacity at zero degrees C. was  
24 less than specification.

25 Increasing the pre-charge to four ampere hours of

eb15 1 oxygen, we again were marginal on measurements. This is  
2 fairly -- not directly after the pre-charge operation but down-  
3 stream a little bit; a fairly marginal, approximately zero  
4 measurable pre-charge. However, the capacity was quite satis-  
5 factory, up in the order of 125 percent of nominal at room  
6 temperature, 25 C. and 110, 115 percent, on that order, at zero  
7 degrees C.

8 Overcharge at zero degrees C., extremely uniform  
9 voltages at C. over 20, something on the order of 400 percent  
10 of capacity input; the first 120 percent or 200 percent goes  
11 in at about C over 10 at 25 and then the temperature is lowered  
12 to zero and continued at C over 20.

13 So we found uniform voltage characteristics on  
14 over-charge, and we do have the over-charge test, and capacity  
15 improved with the increase in pre-charge.

16 Then we had some mavericks which, although we did  
17 some tests on them and I believe that the oxygen pre-charge  
18 is on the order of 6 ampere hours, and those cells had the  
19 highest capacity, again compared to the others.

20 But the interesting thing was that the highest  
21 capacity cells had the least variability in capacity. Maybe  
22 we don't have a large enough sample yet but it appears to be  
23 that there is a variability of capacity factor, also, involved.  
24 You may get some negative limiting influence so you're not  
25 really measuring your positive in every individual cell due to

eb16 1 the variability of pre-charge perhaps or the individual nature.

2 It takes a lot of effort to look into these type  
3 things.

4 I might make one other comment:

5 Going back a while when I was a manufacturer, not  
6 on the user end of the stick, or working in the manufacturing  
7 area, we were able to use a technique to measure pre-charge  
8 after a starved reversal to one volt and that is by oxygen  
9 addition, pressurizing the cell with oxygen and observing  
10 recombination.

11 Unfortunately, it took something on the order of  
12 20 days or more before it began to indicate we were not having  
13 any significant oxygen recombined, but it might be simpler than  
14 a chemical analysis.

15 FONT: Simpler, but perhaps less accuracy.

16 DUNLOP: Jim Dunlop, from Comsat.

17 Dr. Font didn't include some data here that we had  
18 fed in in response to something he said.

19 In the program he actually tried three different  
20 ways to do this before he set the pre-charge and one of them  
21 was to put a resistor across the cell. One of them was to run  
22 it down at a C over 10 rate like you described. And one of  
23 them was just to quit at one volt.

24 Now the reason I make all this-- I'm just saying  
25 the reason we didn't present the data is that in essence, it

eb17 1 does make a difference-- Like you point out, where you put  
2 a one ohm resistor on something and take it all down, it does  
3 take out some capacity, so if you do this, it would change  
4 your pre-charge measurement that you would measure electro-  
5 chemically.

6 The point is here that it didn't really signifi-  
7 cantly change any of the-- In other words, what we do anyway  
8 is completely discharge the cell on down to minus .5 a volt  
9 before we do the analysis and in discharging the plates on down  
10 to minus .5 volt and then doing the analysis -- and that's a  
11 very important point -- in that case, there wasn't any dif-  
12 ference.

13 The number you mentioned, which was three or four  
14 ampere hours or five ampere hours of charged cadmium remaining  
15 after that plate had been completely discharged electro-  
16 chemically, was there whether we use method A, B, C, or D.

17 CARR: Earl Carr, Eagle Picher.

18 I would like to interject one more thing. I think  
19 we had the same or a similar experience and that is that with  
20 the one-ohm load placed on the cell prior to the reversal to  
21 determine pre-charge, we had tests which indicated no pre-  
22 charge and yet we were somewhat confident that there was pre-  
23 charge there.

24 The test on a limited number of cells was run  
25 without putting the one-ohm load and in this case continuing

eb18

1 the discharge in the starved state at a C over 2 rate-- You  
2 mentioned you used C over 10 -- on to zero volts and then on  
3 to minus one volt, and we got the pre-charge that we had in-  
4 tended to put in the cell.

5 So what we have done -- and this is on the SMS  
6 program with Philco-Ford -- is we have concurred that we will  
7 do the pre-charge measurement under these conditions; in other  
8 words, starved, going at C over 2, all the way through zero and  
9 to minus one volt.

10 FONT: In your measurement to minus one volt, do  
11 you take into account the positive capacity in the negative  
12 plate?

13 CARR: I don't know the answer to that. No.

14 MAURER: Maurer, Bell Labs.

15 The negative plate, because of corrosion, has a  
16 substantial positive capacity, nickel capacity; by "substan-  
17 tial," several percent, maybe 10 percent, depending on what  
18 impregnation method is used. And if these negative plates are  
19 reversed during their formation at all, that material becomes  
20 reverse-charged or converted to nickel plus three.

21 Then if you make a cell from those plates and  
22 start to charge, the first thing you do is charge the nickel  
23 and you charge your positive electrode and you charge the  
24 nickel part of your negative electrode and so you build in an  
25 automatic reduced negative charge which you will observe on



ebf9

1 the subsequent discharge as a negative limiting cell.

2 So what you need to do is on your last formation  
3 cycle, stop before you reverse the electrode at all.

4 CARR: When you say "referse the electrode,"  
5 reverse it against what?

6 FONT: Against the counterelectrode.

7 MAURER: Against the counterelectrode.

8 CARR: Okay. Well, we don't do that under normal  
9 circumstances so that we don't think that we have reversed the  
10 negative.

11 MAURER: Well, if you are reversing your cell in  
12 the cell state now to look at pre-charge and then charge it  
13 back up again, you'll go through this problem all over again.

14 CARR: Right. But on the first time down, measur-  
15 ing pre-charge, you would not.

16 MAURER: No, unless you had done it in formation.

17 BETZ: Betz, Fairchild.

18 Just to comment, between Carr and Jim Dunlop, and  
19 that is that the difference in measuring techniques has a  
20 large tendency to affect the data so that when we all talk the  
21 same language I think the standardized test technique-- Really,  
22 we have one but if it is not the best then another one should  
23 be evolved so that when we come to talk, we can all talk the  
24 same language.

The other comment, that I didn't mention, is that

Feb 20 1 I'm sure that we had the same residual initial material from  
2 formation in our cells, and that this is something else.

3 CARR: Earl Carr, Eagle Picher.

4 I concur, Fred, very much. The thing that I was  
5 going to say but I forgot a minute ago and that was the reason  
6 we selected C over 2 was that in this particular application,  
7 which was synchronous orbit at a 60 percent depth of discharge,  
8 this is the rate. So we thought well, this is as close to  
9 being representative of whatever we're going to do while we're  
10 in orbit, and so therefore we're going to use these conditions,  
11 starved, C over 2.

12 We don't put one ohms on them in orbit.

13 MAURER: Maurer, Bell Labs.

14 Another effect I wanted to mention was that a  
15 positive electrode is diffusion limited. Once the voltage drops  
16 below about -- well, below about let's say two volts with  
17 respect to mercury oxide reference, it becomes diffusion  
18 limited and follows the diffusion limiting discharge; that's  
19 diffusion of the protons in the lattice.

20 And this is a very long time process and if you  
21 put a resistor across the cell, as you know, the voltage that  
22 you measure on the resistor indicates a substantial current  
23 still flowing that lasts for, well, the order of 10, 12 hours  
24 at values perhaps above 5/100th of a volt, and it continues  
25 down in a logarithmic fashion which looks like a series --

eb21 1 exponential expansion with slopes in the ratio of 1 to 9 to 27,  
2 and so on.

3 And the capacity available under these conditions  
4 is substantial, perhaps 10 percent of the theoretical capacity  
5 of the electrode, so that if you discharge electrochemically  
6 below one volt on the cell and then immediately reverse, there  
7 will be this amount of positive capacity still there, and that  
8 will be this charge material on the negative.

9 If you put a resistor across you need to leave the  
10 resistor on for a long enough time, perhaps 24 hours, to get  
11 all of this material back out.

12 RANPEL: Guy Rampel, General Electric.

13 I think the important part of this thing here in  
14 measuring the negative capacity residual charge is to say that  
15 the whole thing really is relative and that it depends -- no  
16 matter when it's done, it depends on the amount of cadmium that  
17 is available, because there is a ratio down below, an equilibrium  
18 between active and inactive cadmium, and this equilibrium shifts  
19 from one direction to the other, depending upon plate design,  
20 previous load temperature and so on.

21 So I think that the electrochemical measurement of  
22 negative pre-charge can be viewed as a measurement indicative  
23 of what is available then and there, but it should not be used  
24 as a quantitative measurement to indicate anything except the  
25 part of the cadmium that happens to be available at that moment.

eb22

1 MAURER: There ought to be comments on that.

2 BETZ: Just a quickie.

3 In response to that, that's the way we look at it.

4 In our specification we spec uncharged excess negative but in  
5 order to determine that you've got to get a measure of pre-  
6 charge somehow and the electrochemical technique is used to  
7 reverse the cell, then recharge, and then a measure -- then  
8 recharge flooded and a measure of the total negative. So you  
9 can get a measure of excess uncharged.

10 RAMPEL: Fred, going beyond the reversal and then  
11 adding electrolyte to do the flooding measurements, there are  
12 two problems there, too-- Well, there's another one down below.  
13 Let me get to that one first, and that is rate dependent.

14 If you go, for example, at C over 10, like SAFT,  
15 you run into a serious danger of shorts and parallel resistances  
16 which can throw that whole result off considerably and you can  
17 find more cadmium in the cell than you have theoretically in  
18 some cases.

19 I only point that out to indicate that there are  
20 problems there, too, and maybe C over 2 is a little bit better,  
21 but nevertheless it's still relative.

22 Now going the other way, when you want to find out  
23 how much positive electrodes you have, you flood the cell and  
24 then according to NASA you charge at C over 10 for forty  
25 hours. Okay, that's good but depending upon the activity of

eb23

1 the cadmium electrode you can charge the negative electrode  
2 perhaps, or you think it may be fully charged when you get to  
3 152 but there is some error there, also.

4 As a matter of fact, we have found it absolutely  
5 necessary to take the cell apart for that part of the analysis.

6 Now the next thing I want to point out is now you  
7 are taking a sealed cell and you're flooding it and you're  
8 getting a positive capacity under flooded conditions. Now if  
9 you run ECT or that kind of data flooded, you know that  
10 generally .82 percent -- 82 percent of that can be counted  
11 upon for a sealed capacity, sealed positive capacity. So that  
12 ought to be taken into account also.

13 BETZ: I think your comments are well taken. The  
14 test is not necessarily an absolute measure of the things that  
15 are happening.

16 Incidentally, I found, oh, somewhere around 40 or  
17 50 ampere hours at C over 2 in reversal, also, in a nine ampere  
18 hour cell.

19 BAER: Baer, Goddard.

20 I guess this could be directed at Guy.

21 You said you ran -- checked the electrode capaci-  
22 ties; you ran it flooded. How about if you ran it starved in  
23 that condition also? If you ran the pre-charged starved and  
24 also the check the electrode capacity, if you ran that starved?

RAMPEL: Well, I think we're probably getting away

eb24 1 from the true capacity of positive electrode anyhow, but that  
2 is not at room temperature and as Dean Maurer pointed out this  
3 morning, the charging efficiency of the positive electrode,  
4 besides being related to charge rate, it's also temperature-  
5 related and you have to take these things into account, so  
6 there is no use kidding ourselves.

7 I think we had better just get the absolute amount  
8 of positive capacity that you can get and take that into  
9 regard. I think if we do it sealed we're going to get a lower  
10 value which is fine, but we'll be kidding ourselves.

11 SCOTT: Scott, from TRW.

12 I think underlying some of this discussion is the  
13 presence of an electrochemical procedure for measuring pre-  
14 charge and so forth as it now is in print in the NASA interim  
15 hi-rel spec. From what I hear, I think there is cause to  
16 possibly reconsider that procedure and possibly even set it  
17 aside until -- pending further work, until we know more about  
18 exactly what we're measuring.

19 The other point I have is that also what I hear is  
20 the possibility that the only incontestable measure that we have  
21 right now that we know of of pre-charged cadmium is the chemical  
22 analysis and if that is so, I am wondering whether -- what the  
23 relationship is between a chemical determination of excess  
24 negative and electrochemical performance or expectation in the  
25 cell.

25

1 I don't know what that is. I wonder if  
2 anybody could comment on that?

3 DUNLOP: It may be my procedure that is in that  
4 spec; I'm not really sure whether it is or not.

5 VOICE: No, it's mine.

6 (Laughter.)

7 DUNLOP: Are you talking about chemical analysis,  
8 Dr. Scott?

9 SCOTT: No, I thought you were referring to the  
10 electrochemical --

11 DUNLOP: We were talking about chemical analysis  
12 here a minute ago.

13 SCOTT: And I thought you were just referring to  
14 the electrochemical procedure in the NASA spec.

15 DUNLOP: Unfortunately I don't know what's in the  
16 NASA spec right now. The last NASA spec I looked at didn't  
17 have-- I don't remember that it really mentioned a measure-  
18 ment procedure for pre-charge, frankly.

19 I'm not really at all clear what you're  
20 talking about.

21  
22 SCOTT: I'm talking about the original interim spec  
23 issued in '69 or '68.

24 DUNLOP: The procedure for discharging down to  
25 minus half a volt; is that what you're talking about?

eb26

1 SCOTT: Yes. That's the one that I think people  
2 are still trying to use because it is there and it had a certain  
3 degree of legitimacy because it's there, --

4 DUNLOP: Okay.

5 SCOTT: -- regardless of whether it is any good or  
6 not.

7 Okay?

8 DUNLOP: Okay.

9 SCOTT: That's the one I was referring to.

10 DUNLOP: Okay. I just wanted to clear that up.

11 Thanks.

12 FORD: Ford, NASA/Goddard.

13 Then we still have a basic question: Why can't  
14 you measure pre-charge electrochemically? What is it in the  
15 cell that prohibits you from doing this?

16 I have had it suggested that this is influenced by  
17 the mechanical pressure exerted within a cell.

18 MAURER: Maurer, Bell Labs.

19 I'd like to suggest a method that we've used and  
20 that is to reverse the cell and measure the gas coming off,  
21 confirm that it's hydrogen, and measure its rate. And then  
22 when the cell voltage goes over the knee, indicating that it  
23 would appear that the negative has completely discharged, then  
24 you compare the electrical current or capacity back through  
25 with the amount of hydrogen that you've collected. If they



eb27 1 agree, I think you can be reasonably sure that that was the  
2 pre-charge.

3 However, if they don't agree, then you're in trouble.

4 (Laughter.)

5 HALPERT: Halpert, of Goddard.

6 In response to Floyd's question, we've got to learn  
7 how to measure it, I think we've got to learn how to put it in  
8 before we can learn how to measure it, put it in reliably and  
9 uniformly every time.

10 BELOVE: Belove, Marathon.

11 The question there, Gerry, would be how do you  
12 know what you're putting in, if you can't measure it.

13 (Laughter.)

14 But I have another point here. Do you have to  
15 have the absolute measurement of pre-charge? I believe that  
16 any method you use is only going to be an approximation. The  
17 electrochemical tells you how much cadmium you have, but again  
18 we have this question of percentage of inactive cadmium and  
19 a certain amount of it in the form of cadmium hydroxide.

20 In every way you measure it you will have certain  
21 factors, as Guy pointed out, the rate of discharge, the  
22 temperature. All of these factors can come into the absolute  
23 value that you determine, and what I say is that this is not  
24 necessary.

25 What is necessary is to obtain a relative -- an

eb28 1 accurate measurement of the relative amount of pre-charge and  
2 to see what effect this has upon cell performance.

3 SCOTT: Scott, TRW.

4 I wonder if this afternoon we cannot come to one  
5 tentative conclusion, or whether we cannot, that electrochemical  
6 measurement is not likely to give us a reproduceable measure  
7 of pre-charge. And then we can go on from there.

8 I've heard several comments to the effect that  
9 variations on maybe one certain way are able to measure pre-  
10 charge reliably. I'm not sure whether that's the message or  
11 not. But I wonder if somebody would want to comment on that?

12 DUNLOP: Let me comment. I'm sitting here kind of  
13 like a cat on a hot tin roof because I'd like to go up and  
14 make a presentation.--

15 (Laughter.)

16 -- which is very relevant to all this discussion, frankly,  
17 because we've been making these measurements for two years and  
18 we'd like to show what we have, and I think they might lend some  
19 light on this whole discussion as to what -- how repeatable  
20 these type of measurements can be.

21 And frankly, to tell you the truth, we've been  
22 measuring GE cells and the measurements we get show that the  
23 results are very consistent. As a matter of fact it shows their  
24 process is in very good control. As a matter of fact it shows  
25 that this method of running it in reversal gives you very

1 repeatable results after about the first 30 or 40 cycles.

2 MAURER: Let's get on with the data and save some  
3 of the philosophy until later then.

4 Jim will give a dissertation on some of his  
5 measurements.

6 DUNLOP: Dr. Font just got done describing to you  
7 about an hour ago --

8 (Laughter.)

9 -- his method for adjusting the pre-charge at three different  
10 levels, measuring it and what the effects were initially and  
11 after some 30 cycles on the pre-charge in terms of the pressure  
12 level and ampere hour capacity.

13 I might say that the reason that we entered into  
14 this contract starting about a year ago, I guess, was that we  
15 wanted to evaluate exactly what Dr. Font has done, what SAFT  
16 has done, and we're very pleased with the results. And I  
17 think finally-- You know, the problem for a long time was  
18 that nobody would really discuss this very openly because it  
19 was in some kind of a proprietary category or something, and now  
20 it seems that everybody wants to discuss it in depth.

21 We planned to take those cells from SAFT with the  
22 different pre-charge levels and operate them in synchronous  
23 application and try and determine what the effects of the  
24 different levels are over long-term periods of time. What  
25 they have done is show what the effects are over a short period

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eb35

1 of time.

2 We have as a background for this program a consider-  
3 able amount of experimental data from our Intelsat-4 effort.  
4 We started a program in 1969 to simulate the Intelsat-4 mission.  
5 with the only variable being this storage mode. We picked  
6 three different storage modes at that time.

7 The storage modes we used were trickle charged  
8 continuously during storage; open circuit charged with re-  
9 conditioning every 30 days; and open circuit discharged.

10 Now we decided in the course of that program that  
11 we'd pull a cell out from each of these storage modes every  
12 six months, every eclipse season, and analyze that cell and  
13 we had to pick a couple of years ago the procedure that we  
14 would use to analyze it.

15 At that time, the procedure we used was the pro-  
16 cedure that was recommended by Dr. Flasher, being used at  
17 Battelle for analyzing the negative. The only thing that we  
18 added to that procedure, frankly, was our approach to doing  
19 in what we consider a little more consistent manner. And I  
20 don't necessarily want to go into the fine points.

21 We have documented that procedure and described it,  
22 I thought in some detail, last year. Gerd Van Ohmring and I  
23 made our presentation last year along with Dr. Parry who  
24 described the positive analysis that he was using at that time.

What I'd like to do today is show some of the

eb31 1 results from that program. And when I show these results, I  
2 want to say that I believe, based on these results, that we are  
3 getting ourselves in the position of being able to make some  
4 measurements which do make sense and can be used to evaluate  
5 or estimate what the lifetime expectancy of those cells is going  
6 to be in a synchronous application and that under the type of  
7 operating conditions we are pretty sure we expect to see in a  
8 synchronous application.

9           It may or may not be germane or relevant to the  
10 30-60 minute cycle.

11           With that in mind, I am going to start with my  
12 first graph here.

13           (Slide 179.)

14           The data that I'm presenting here is data on the  
15 pre-charge cadmium variation with time determined for cells that  
16 were trickle charged during the storage mode.

17           The zero reference point here is actually a  
18 measurement made when the cells have gone through a normal ac-  
19 ceptance testing program either at Hughes Aircraft or at Comsat  
20 so they have already been through at least 30-60 cycles. At  
21 that point in time we found that all the cells that we've looked  
22 at -- and I'm only showing three on this graph, but we must have  
23 looked at approximately 10 or 20 with this method. In all the  
24 cells that we've looked at so far, the total pre-charge

25 determined by both chemical and electrochemical analysis, has

eb32 1 been 12 ampere hours plus or minus one ampere hour.

2 Now I think that indicates that their process is  
3 in pretty good control when they set the pre-charge.

4 The amount of pre-charge determined electro-  
5 chemically is also fairly uniform at this point in time. It has  
6 been about six ampere hours plus or minus one ampere hour  
7 determined electrochemically. That's the electrochemically  
8 measured pre-charge at a C over 2 rate, no one-ohm resistor  
9 or anything else.

10 And we use the C over 2 rate, just like everybody  
11 else does, because that's the way we're running them in orbit.  
12 We're using about a C over 2 rate, so we discharge it down  
13 right through on reversal. We puncture the cell and we also  
14 measure the gas so that the amount of gas that escapes has to  
15 correlate with the number of ampere hours that we measure.

16 After we completely discharge that cell to half a  
17 volt we cut it open with that famous Goddard cutter and put  
18 it in a nickel soxlet and extract the electrolyte, and then  
19 we take the plates out, dry them, and perform the famous  
20 muskrat analysis to determine how much charged cadmium is  
21 remaining.

22 The charged cadmium remaining has been something  
23 between 5 and 7 ampere hours at this point in time so that the  
24 total, adding together the amount of charged cadmium remaining  
25 and the amount of charged cadmium measured electrochemically

eb33

1 comes out to be this 12 plus or minus one ampere hours.

2 Now the interesting thing about this data is that  
3 we continued doing this analysis after one eclipse season, two  
4 eclipse seasons, three eclipse seasons and up to four. That  
5 represents cells on test since two and a half years now, and  
6 what we are observing is that after one eclipse season, the  
7 amount of electrochemical measured pre-charge increased from  
8 the initial value of about six up to about eight. In the second  
9 season it was still about eight. In the third season it was  
10 still about eight; actually it was dropping off a little bit,  
11 but this kind of variation, seven and a half to eight.

12 So that the amount of pre-charge that we are measur-  
13 ing up to two years now electrochemically is fairly consistent  
14 for the cells that we're looking at.

15 And if we take the amount of pre-charging that we  
16 measured electrochemically -- Each time we also determine the  
17 amount of pre-charge from a chemical analysis and we continue  
18 to come up with-- Actually it looks like this pre-charge is  
19 increasing slightly with time.

20 Now we actually expect that to happen because of the  
21 level of carbonate buildup that we're observing in that cell,  
22 and this line that I put on right here is a line which just  
23 relates the level of carbonate buildup that we're observing  
24 with time to what we would expect that to do in terms of chang-  
25 ing the pre-charge level.

eb34- 1           These particular cells are made with the pella  
2 separators.

(Slide 180.)

3           This data shows the carbonate buildup observed with  
4 time. At the same zero point in time or after the cells have  
5 been through the 30 cycles of burn-in, we measure about five  
6 grams of carbonate in that electrolyte and we measure five  
7 grams of carbonate -- it's really about five and a half, and  
8 it's very close-- All the cells that we've analyzed have some-  
9 thing very close to around five or five and a half grams of  
10 carbonate initially.

11           As a matter of fact, the SAFT cells have a carbonate  
12 level in their cells, too, and percentage-wise it is almost  
13 the same. I really don't know why they have this problem but  
14 I think this has been discussed earlier and I think it is  
15 coming from the plates.

16           We notice that as we run these cells and do this  
17 analysis again six months later -- The X's here represent cells  
18 that are trickle charged -- you will notice that the carbonate  
19 level is increasing; you're up to about-- After one eclipse  
20 season, about six and a half, seven. You're up here about seven  
21 and a half, eight, or seven and a half grams of carbonate with  
22 time..

23           Now there's some spread on this data, obviously,  
24 but you do see that there is the increase in the carbonate  
25 level for these cells and that if you go to a different storage



eb35 1 moe , if you go to a storage mode where you're not continuously  
2 charging, where you only have an oxygen environment or your  
3 oxygen environment in your cells is much lower, or as Dean  
4 Maurer pointed out, there is maybe not a chemical-reducing  
5 effect but an electrochemical reducing effect-- I didn't hear  
6 that paper but I heard about it yesterday.

7           Either way, you would expect that the cells that  
8 were trickle charged continuously would have a higher rate of  
9 carbonate buildup with time.

10           Based on these results we sort of estimate that if  
11 you are storing cells with a pella separator in a trickle charge  
12 mode, you are going to have a buildup of between 1.4 and 2 grams  
13 of  $K_2CO_3$  per year in these cells. If you relate that-- You  
14 really need to relate that to the total amount of electrolyte  
15 in your cell.

16           I believe that the initial carbonate level on a  
17 percent weight basis is about 25 percent of the KOH and so you  
18 can get an idea. At this point in time we're up to about 30,  
19 35 percent by weight carbonate of your KOH.

20           (Slide 181.)

21           Again, in this test program we were running we were--  
22 As we analyzed these cells after we got done taking the cells  
23 apart, we did take some of the plates and run them in a flooded  
24 bath to determine what their utilization was.

25           You will notice the utilization is running-- It

eb36

1 starts out as high as 80 percent when we first receive them;  
2 it drops down to about 75 percent after the burn-in; after  
3 the first eclipse season it's to 71, 72, 73 percent, 71, 72,  
4 73, 69 percent.

5 So most of the change in utilization appears to be  
6 occurring in the first six months to a year and thereafter,  
7 these cells appear to have, at least in the flooded electro-  
8 lyte, a very consistent utilization of the cadmium material  
9 and it's about 70 percent.

10 Now that number is not too different from the  
11 number that Dr. Maurer presented this morning. I think he was  
12 showing about 74, 75 percent utilization for the Bell Labs  
13 plates.

14 By the way, I want to make one more comment here.

15 This utilization that we get here is pretty good  
16 and I do think that Dean Maurer has possibly the right idea.  
17 To get this kind of utilization I do think that you do need to  
18 exercise and overcharge that cell somewhat. And the reason I  
19 say that is that of the three storage modes that we have -- and  
20 I'm not going to present all of the data today -- but the one  
21 mode that we threw out already was the mode in which we stored  
22 the cells passively during the entire eclipse season. And  
23 those cells, by the third eclipse season, started to generate  
24 overvoltage potential and indicating no overcharge protection,  
25 indicating that you couldn't utilize that cadmium for overcharge

eb37

1 protection when you were storing those plates or those cells  
2 passively.

3 But the other two storage modes, the one in which  
4 you exercised it every 30 days and the one in which you con-  
5 tinuously trickle charged, we have almost identical voltages  
6 at the end of charge, identical voltage performance from dis-  
7 charge, and they seem to have very similar characteristics,  
8 and I think we have very similar utilization of the cadmium,  
9 too.

10 So it does require some amount of overcharge and  
11 some amount of periodically exercising those plates to maintain  
12 that utilization.

13 (Slide 182.)

14 Finally, in a bar graph fashion somewhat similar  
15 to the bar graph again that Dr. Maurer presented this morning,  
16 we have looked at the cadmium electrode after burn-in, one  
17 eclipse season, two eclipse seasons, and three eclipse seasons  
18 and three storage periods, to determine how the cadmium material  
19 appeared to be distributed.

20 The bottom portion here represents unusable pre-  
21 charge determined from the chemical analysis. The second  
22 block here represents the usable pre-charge determined electro-  
23 chemically by discharging the cell in the reversal and C over  
24 2 rate to minus half a volt.

25 The third block here represents the positive cell

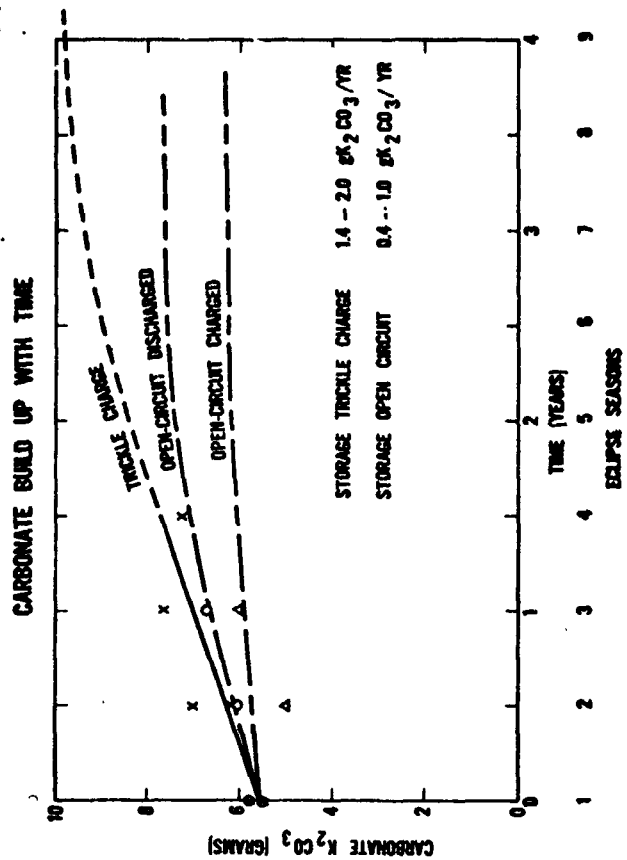


Figure 180

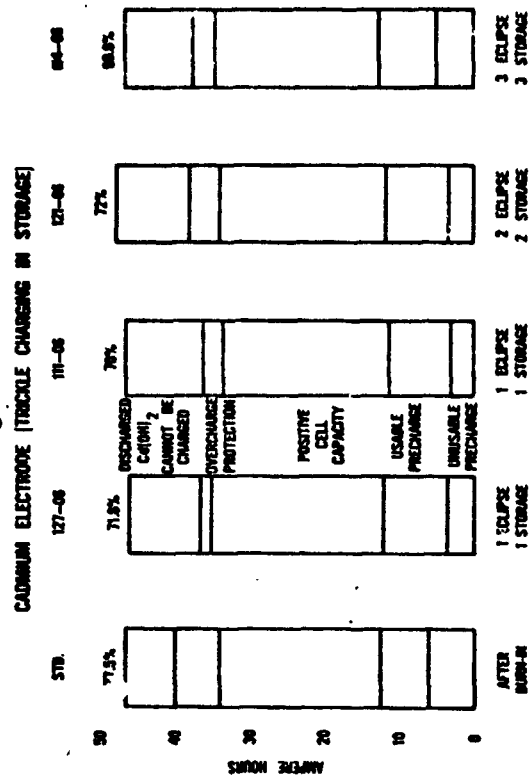


Figure 182

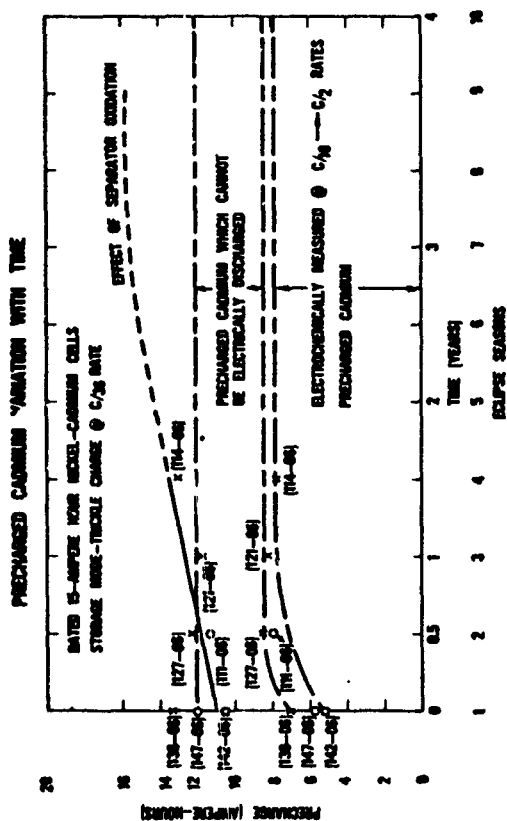


Figure 179

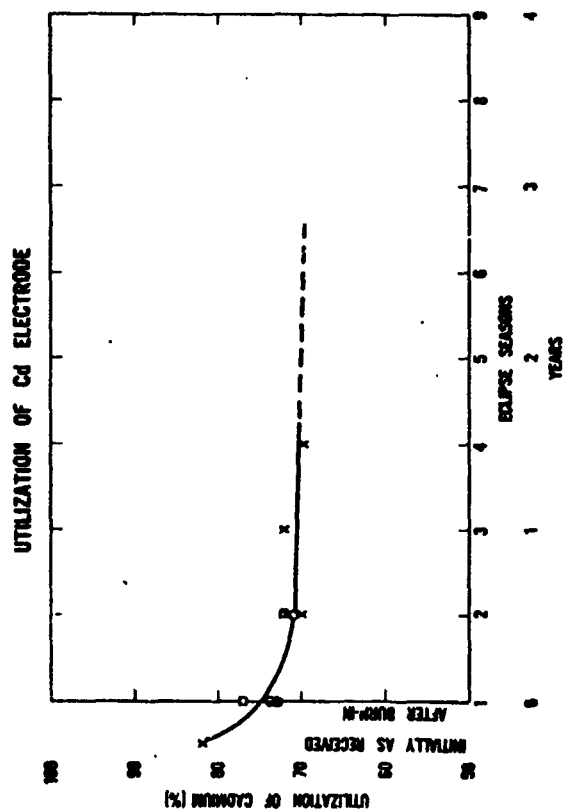


Figure 181

eb38

1 capacity where the portion of the cadmium hydroxide is being  
2 used in cycling.

3 The fourth block here represents overcharge pro-  
4 tection.

5 And the fifth block here would appear to be dis-  
6 charged cadmium which can no longer be charged.

7 The total pre-charge as we define it appears to  
8 remain fairly consistent or be on a slight upgrade here. The  
9 amount of non-usable pre-charge seems to be staying relatively  
10 constant. The amount of usable pre-charge, again relatively  
11 constant; only slight changes.

12 What primarily concerns us for a seven-year mission  
13 is the amount of cadmium which appears to be there for over-  
14 charge protection. We are beginning to make predictions based  
15 on the kind of results that we are observing as to how long  
16 cells will last. We're making these predictions, based on the  
17 kind of measurements that we can take at this point in time and  
18 using data of this type to extrapolate out.

19 What we're predicting is that after one year, the  
20 utilization of the cadmium electrode will be about 70 percent;  
21 that you will have at least 65 percent utilization of the  
22 amount of pre-charge material that you used. After one year,  
23 there will not be a significant change in the pre-charge.

24 So whether you measure it-- If you look at the  
25 electrochemical measurement, it really is immaterial to me

eb39

1 whether you make the chemical measurement at that point in time.  
2 All I'm saying is that after one or two years of time, there  
3 isn't going to be any change here.

4           The amount of overcharge protection is going to  
5 remain about the same after one or two years with one excep-  
6 tion. As you have any method in that cell which is going to  
7 allow either oxidation of the separator as has already been  
8 described, such that there is going to be the shifting in the  
9 pre-charge level, or any method which is going to cause posi-  
10 tive method to increase due to attack of the nickel, as  
11 Dr. Font described, or any other slight change in the amount  
12 of cadmium hydroxide which becomes non-usable, as Dr. Maurer  
13 described, all these procedures tend to cut into the over-  
14 charge protection.

15           None of them really affect anything down here but  
16 all of them affect the amount of overcharge protection.

17           We estimate that after you get by one or two years  
18 that you need about one to two percent per year cadmium over-  
19 charge protection so from that kind of an argument you can  
20 hypothesize how much overcharge protection you need to get to  
21 four-year life, five-year life, and six-year life.

22           On top of that one or two percent you've got to  
23 have some variation due to the amount of cadmium that you have  
24 in there or the amount of pre-charge that you put in there  
25 and as I have pointed out, in these particular cells that

1 variation is relatively small.

2 In summary, I'd just simply like to say that we  
3 would like to see in a specification some type of procedure  
4 standardized to be used to evaluate cells for synchronous  
5 application and I believe that we, based on the kind of infor-  
6 mation that we presented here, we are getting closer to being  
7 able to make that kind of a prediction based on the kind of  
8 test program that we're proposing to run.

9 In essence, what I'm saying is that I think there  
10 can be a test written that will answer the question of how  
11 good is a cell and how good a chance does it have in meeting  
12 a five-year life, based simply on the type of measurements  
13 that you can make when you receive the cell and run it through  
14 a 30-cycle burn-in test.

15 STEINHAUER: Steinhauer, Hughes.

16 Jim, I'd like to comment on the discharge storage  
17 mode in that I think you can't use it without some low rate  
18 reconditioning. The question I would like to ask is:

19 I think I've been listening carefully but Dean  
20 Maurer made a statement this morning that inactive cadmium is  
21 predominantly if not all in the charged cadmium mode. You have  
22 exhibited a very large portion of inactive material in the  
23 cadmium hydroxide upstairs.

24 Do we have a controversy here or did I hear wrong?

25 DUNLOP: We have a controversy to some degree. I

eb41E

1 think you saw what Dr. Font presented. Now let me review that  
2 for a moment.

3 When we went through the formation cycle, the  
4 results we got in that program I think are very similar to the  
5 kind of results we got from any other formation cycle. In  
6 other words, we get about a 1.7 to one ratio of negative to  
7 positive materials.

8 That 1.7 to one ratio of negative to positive means  
9 that there is not enough completely electrochemically utilizing  
10 that negative plate at that time because you're putting into  
11 that negative plate about a two to one ratio.

12 When SAFT does the analysis they show a two to one  
13 ratio. They show that in that 72 ampere hour plate, for  
14 example, that Dr. Font described, there were three or four  
15 ampere hours of charged cadmium that couldn't be discharged  
16 in the analysis and there were six to eight ampere hours of  
17 discharged cadmium.

18 Just using those numbers, that means that there's  
19 something like five, six, or eight ampere hours of discharged  
20 cadmium initially in the formation cycle which cannot be  
21 utilized.

22 Dr. Maurer this morning made a statement that if  
23 you converted some of this cadmium in a charged state to  
24 discharged cadmium, then it may become nonusable. A portion of  
25 it or all of it will become nonusable.



eb42 1 STEINHAUER: Was that a firm statement or an un-  
2 certainty?

3 MAURER: That's an uncertainty.

4 FORD: Ford, NASA/Goddard.

5 Jim, in your-- You indicated your overcharge  
6 capability had diminished because you cadmium you could not  
7 charge. Have you tried to find out why it can't be charged,  
8 what form it's in.

9 DUNLOP: Well, the analysis says it's discharged  
10 cadmium that can't be charged. You know, it's cadmium hydro-  
11 xide that can't be charged.

12 FORD: Why? What ties it up?

13 DUNLOP: Of course there are a lot of arguments  
14 here. We don't know the right answer. There were some pic-  
15 tures that Halpert showed this morning, very interesting. He  
16 showed, for example, some kind of a glob -- excuse that word --  
17 of cadmium hydroxide and cadmium in which he showed that-- He  
18 pointed to the darker and lighter areas and he showed that the  
19 outside of this ball was cadmium hydroxide and the inside was  
20 cadmium.

21 Now, if that were the case you would not be able  
22 to discharge that cadmium inside there. That would explain  
23 discharged cadmium that could not be charged.-- I said it the  
24 wrong way.

25 SCOTT: Scott, TRW.

eb43 1 At the risk of being repetitious, I don't think  
2 that I caught your experimental method of distinguishing in-  
3 active from active discharged cadmium. Analytically, how do  
4 you do that?

5 DUNLOP: Do you want to know the discharged cadmium?  
6 How I did it on-- Okay.

7 SCOTT: You have a bar chart showing-- At the top  
8 you have two forms of discharged excess cadmium or discharged  
9 cadmium. How do you experimentally determine those two blocks?

10 DUNLOP: Okay. For all the data that is presented  
11 here we started our analysis by fully charging or charging at  
12 a C over 10 rate for 16 hours the cell, then discharging it  
13 down, running it in reversal, and then opening it up.

14 We then took the cell apart and measured the  
15 flooded capacity of the negative plates and we assumed -- and  
16 this is, by the way, if anything I think probably an optimistic  
17 assumption, that that represents the amount of cadmium that can  
18 be -- The flooded plate electrochemical measurements indicate  
19 the amount of cadmium that can be used in the cell.

20 If that's the case then you simply go from here to  
21 here. That's the difference from here to here.

22 Now if the actual dry -- in the starved condition --  
23 utilization of the cadmium is less than that, this level is  
24 going to move down. What we intend to do next time-- We've  
25 had this question raised before, so what we intend to do next

eb44 1 time around is sacrifice the measurement of the pre-charge  
2 by charging the cell up and then venting oxygen and measuring  
3 the quantity of oxygen that we vent until we get to an over-  
4 voltage condition, and do it on that basis to confirm the  
5 measurement that we are currently making.

6 GROSS: Sid Gross, Boeing.

7 Jim, you said you had three methods of charge,  
8 three methods of storage, two on open circuit and the one that  
9 was open circuit discharged didn't charge up very well whereas  
10 the one that was open circuit charged and reconditioned  
11 periodically worked out all right. I presume that's correct?

12 DUNLOP: No. What I said was that the one that was  
13 stored discharged developed high voltage characteristics during  
14 the eclipse cycle, went to overvoltage, went over a 1.5 volts,  
15 went up to 1.5 volts at 75 degrees F.

16 GROSS: And the one that was open circuit charged  
17 and periodically reconditioned, how did that work out?

18 DUNLOP: Fine.

19 GROSS: Okay.

20 Do you attribute this to the discharge part of the  
21 conditioning process or to the overcharge that probably followed  
22 it?

23 DUNLOP: I attribute it to the fact that we were  
24 storing the cell almost completely discharged. In other words,  
25 this would all be cadmium hydroxide and when we tried to

45 1 utilize the cell after normal reconditioning by the normal  
2 procedure that we proposed to use -- that's by a couple of  
3 cycles of reconditioning before the eclipse, but during the  
4 eclipse season by the 30 or 40 cycle -- over 50 percent of  
5 these cells were beginning to go to -- had pulled out from  
6 what the other cells with the other storage modes overvoltage  
7 potential was had pulled out to the hydrogen evolution poten-  
8 tial.

9 GROSS: Well, I understood you to say open circuit  
10 charged for that second mode. Was it open circuit charged or  
11 open circuit discharged?

12 DUNLOP: We had an open circuit charged which we  
13 reconditioned every 30 days. That mode is behaving okay.

14 We had open circuit discharged with no recondi-  
15 tioning, or what I mean by that, no conditioning during the  
16 storage period. We didn't condition it every 30 days; we  
17 reconditioned it before going into the eclipse.

18 RAMPEL: Guy Rampel, General Electric.

19 On the storage mode that gave you overvoltage, I  
20 think you said that in that particular case the cadmium hydro-  
21 xide that was there was not available.

22 DUNLOP: It would appear so because we generated  
23 high voltages.

24 RAMPEL: Okay. So likewise on the upper bar chart  
25 there where you indicate discharged cadmium hydroxide which

eh46

1 cannot be charged.

2 And now, going back to the discharge mode of  
3 storage, apparently as we are all aware, the cell can be re-  
4 conditioned and that cadmium hydroxide be made available again,  
5 so what I wanted to suggest --

6 DUNLOP: Guy, you're talking about a different mode  
7 of storage. If you're talking about shorting a cell with a  
8 wire, that's a different mode than leaving the cell discharged  
9 open circuited. That's a rather fine point but it is a little  
10 different.

11 RAMPEL: Okay. What I'd like to suggest though  
12 nevertheless is that there is an equilibrium of available  
13 cadmium down below between the active and inactive and perhaps  
14 at the top there is likewise an equilibrium situation.  
15 Particularly the charts we're looking at right now determined  
16 statically flooded and in the sealed cell, there may be a ratio.  
17 The availability may shift.

18 SCOTT: Scott, TRW.

19 Jim, you're talking about this discharge storage  
20 mode and the results you got when you started cycling that in  
21 a normal eclipse season. Did you-- Have you taken apart  
22 cells stored in that mode and then measured the actual amount  
23 of discharged negative material or characterized it in any  
24 other way to corroborate your theory that it's inactive dis-  
25 charge material.

eb47

1 DUNLOP: We have done that and there are a couple  
2 of things we've found out.

3 First of all, I have only shown here the data that  
4 we have from the trickle charge mode. In the modes where we  
5 store passive we-- Actually you lose utilization of the pre-  
6 charge cadmium, in other words the charged cadmium that can't  
7 be discharged, as well as the discharged cadmium that can't be  
8 charged. I didn't bring any graphs along to show this point  
9 but by the third eclipse season we find, using this same  
10 method of reversing and electrochemically trying to measure  
11 pre-charge that we are only able to measure one or two ampere  
12 hours rather than the eight ampere hours that I'm showing here.  
13 That's the first point I want to make, Dr. Scott.

14 So we're showing that during these passive storage  
15 modes we are not able to utilize the cadmium -- pre-charge  
16 cadmium. That's the first point.

17 The second point is that we're not able to utilize  
18 as much of the discharged cadmium. One of the problems that  
19 we didn't do and that-- See, the correct way to measure this  
20 if you really run into a problem is going to a venting tech-  
21 nique. Then once you already are at the hydrogen evolution  
22 potential, there is no longer any point in going to a venting  
23 technique; you're already at the point that you started venting  
24 to get to.

SCHULMAN: Schulman, Gulton.

548 1 Jim, did you ever attempt to measure the pre-charge  
2 by one-ohming them rather than by discharging a minus .5 at  
3 C over 2 rate?

4 DALTON: No.

5 FORD: Ford, NASA/Goddard.

6 Jim, I can't let you pass one point that you--  
7 Perhaps it was a subtle point but I don't think it is quite so  
8 subtle. In the difference in storage modes where your dis-  
9 charged open circuit are truly stored in a shorted condition,  
10 as was presented last year by Steve Gaston, some experience  
11 on the OAO program comparing cells that had been stored for a  
12 period of one year in a shorted condition, we saw no signifi-  
13 cant change in the overcharge voltage at room temperature  
14 or at zero degrees C.

15 We have seen similar tests recently as was mentioned  
16 yesterday, but we also have some data from the ITOS battery,  
17 two of them, in fact, that was used as backup batteries -- These  
18 were GE four ampere hour cells that were manufactured -- I'm  
19 recalling from memory now, but I think it was early or some  
20 time in 1968. These cells were fabricated, built into batteries,  
21 the cells went through the acceptance test, the batteries went  
22 through acceptance test, and then they were put in a storage  
23 mode.

24 The storage mode called for the cells to be dis-  
25 charged and shorted but it was an oversight and the cells were

eb49 1 left open circuit.

2           This summer we were asked to evaluate those bat-  
3 teries as far as flight worthiness to be used again in a back-  
4 up spacecraft. The only change we observed on those cells from  
5 the initial capacity that we had was the change in the over-  
6 charge voltage at low temperature. Those cells could not be  
7 overcharged at zero degrees C.F. and incidentally we had  
8 manufacturer's data that we were repeating a test that the  
9 manufacturer had conducted at the cell level when he made the  
10 cells.

11           Those cells could not take an overcharge as low as  
12 C over 40. In fact, it is safe to say those cells couldn't  
13 be overcharged at zero degrees C. at C over 100 without exceed-  
14 ing a voltage of 155 volts per cell.

15           So I'd like to point out that I think the storage  
16 mode gets critical even when you talk about discharged open  
17 circuit or a shorted mode.

18           DUNLOP: Well, Floyd, maybe I missed the point.  
19 The discharged cells were the ones that gave us the problem.

20           FORD: Yes, that's the same experience we had,  
21 discharged but not resisted down and shorted out.

22           DUNLOP: I didn't mean to pass that over as the  
23 right point.

24           I wanted to add one thing to Dr. Scott's comment.

25           Let me add one thing more, Dr. Scott, to this comment



eb50

1 about the amount of cadmium that can be used.

2 In the cells that we have stored in the passive  
3 modes, both of those groups, we were not using our normal  
4 method of discharging the cell down. We were only able to  
5 measure one or two ampere hours electrochemically of cadmium  
6 in a pre-charged state.

7 We followed exactly the same analytical procedure  
8 and measured 10 to 11 ampere hours of cadmium remaining in a  
9 charged state from the chemical analysis so that the total  
10 measurable pre-charge adding the electrochemical measured  
11 value to the chemically measured value gave the same total that  
12 we would expect to get.

13 FORD: Ford, NASA/Goddard.

14 One other point I'd like to make-- And let's go  
15 back to the basic question. Why are we trying to measure pre-  
16 charge? Is it in fact to establish the overcharge capability  
17 of the cell? That was the original intent, back when we first  
18 got interested in this.

19 So perhaps the technique is to let's just try to  
20 come up with a technique to measure the overcharge margin that  
21 we have in the cell and forget about measuring pre-charge.

22 DUNLOP: Well, Ford, look, maybe that's fine but  
23 when I buy a cell from a battery manufacturer -- and this is  
24 what I think is germane to the point -- I'm going to specify  
25 a certain positive to negative ratio. I'm going to go ahead

eb51

1 and I'm going to specify a certain amount of that ratio of  
2 excess cadmium that I'd like to have for overcharge protection.

3 Based on what Dean Maurer said, I am also going to  
4 specify at least 10 percent of that cadmium electrode in a  
5 pre-charge state because I don't want hydrogen to be evolved  
6 initially when I start charging and actually we've seen this  
7 kind of problem can happen. You do need a small amount of pre-  
8 charge in there, something like about 10 percent, and that's  
9 exactly--

10 I didn't make any comment here but I think if I  
11 were going to make a cell today and specify today with a posi-  
12 tive to negative ratio of 1.7 to one measured electrochemically  
13 and two to one chemically, I would request that the battery  
14 manufacturer put 10 percent of his cadmium in an uncharged  
15 state so that he has the capability to achieve the seven-year  
16 mission.

17 And I have to do something in order to be able to  
18 determine this, to make this measurement.

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1 There are a number of points being brought up  
2 here, and I would like to spend a moment to expound on the  
3 issue of hydrogen-free capacity on cadmium electrodes.

4 The charging process on the cadmium electrode  
5 consists of cadmium hydroxide going into solution to  
6 cadmium hydroxide ions which are then electrochemically  
7 reduced to cadmium on the electrode. This is a soluble  
8 intermediate step, it's a chemical step and it has nothing  
9 to do with the electrochemistry.

10 So that the voltage, or the polarization on the  
11 negative electrode is controlled not by this stuff but by  
12 the concentration of this in the electrolyte.

13 So that if you have a lot of very small cadmium  
14 hydroxide crystals the quantity of cadmium hydroxide in  
15 solution is high and the polarization for this reaction is  
16 low, and proceeds nicely.

17 When you have big crystals, or when the electrode  
18 is mostly charged, and you have a few large chunks remaining,  
19 the rate at which this reaction can go now will be the rate  
20 limiting one, so that the concentration of this in the  
21 electrolyte will fall and consequently the polarization for  
22 this reaction will go up.

23 Now as it goes up it reaches the point where the  
24 reactions going on at the electrode compete strongly with the  
25 water going to hydrogen reaction, whose polarization is

1 normally lower than this one. --is higher than this one.  
2 But when this concentration drops, this polarization comes  
3 up. And then this one takes over.

4 So that the concept of cadmium hydroxide which  
5 is electrochemically inactive is perhaps a little bit mis-  
6 leading. What we really mean is that it's functionally in-  
7 active; that if you put the electrode in a flooded state  
8 and charged it for a long time, I think you could get it all  
9 charged; there would be no cadmium hydroxide remaining on  
10 the electrode at all.

11 But the point is that most of it would have been  
12 charged -- or some of it would have been charged with the  
13 evolution of hydrogen; which we don't want in the sealed  
14 cell.

15 So that when we've been talking about inactive  
16 cadmium hydroxide, what we mean is cadmium hydroxide existing  
17 in such a state, such as in large crystals; and these could  
18 result from high temperature storage or high temperature  
19 overcharge, or a whole variety of ways; these crystals now  
20 provide too little cadmium in solution to sustain this  
21 reaction without hydrogen evolution.

22 So that's what we mean.

23 This is why it's rather indefinable, or why  
24 measurements of it are many times misleading. Because just  
25 what you call "inactive" depends on just what conditions you

wb3 1 have and what conditions you're trying to measure.

2 If you try to measure it on very rapid charge  
3 characteristics, you'll find you have a lot of inactives;  
4 if you do it very slowly you'll have less. And so on.  
5 So I think we have to bear that in mind.

6 The reaction is: CdOH taken twice, going to  
7 CdOH taken three times, with a minus sign. And then this  
8 is followed by electrochemical step in which Cd-zero is  
9 formed.

10 STEINHAUER: Steinhauer, Hughes.

11 A two-part question. First, Dean, the cells  
12 you described this morning for Bell Telephone service, are  
13 these shallow depth use or deep?

14 MAURER: Well, they're designed for deep,  
15 100 percent discharge.

16 STEINHAUER: I was just wondering, then, on  
17 this amount of pre-charge necessary for long life, how much  
18 that percentage might be. And then I wanted to ask Jim  
19 if he would put that 10 percent in an inactive form or in  
20 an active form?

21 MAURER: I'm not sure that I understand what  
22 the first part of your question is. But we would put about  
23 10 percent pre-charge in an active form. We would start  
24 with the electrode as it's formed in the cell, and remove  
25 enough oxygen to give us an additional 10 percent, roughly.

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1 GASTON: Gaston, Grumman.

2 I have a question to Jim. I believe one of his  
3 first comments was in the pre-charge adjustment that the  
4 procedures were controlled and repeatable.

5 What is the pre-charge adjustment procedure?  
6 I don't believe you commented on that.

7 DUNLOP: I don't want to answer for G.E., and  
8 I really don't know, so I really shouldn't say. But I do  
9 know that they do a certain amount of oxygen venting; and  
10 I don't know how much, and I don't know anything else  
11 about it. But I do believe it's an oxygen venting technique  
12 that they use.

13 And the only point I made was not how it was done  
14 but the fact that our measurements show that the amount of  
15 precharge that they're putting in there is repeatable from  
16 cell-to-cell and from lot-to-lot.

17 GASTON: My comment to that is, I believe we  
18 would like to look for procedures which we can specify,  
19 which are repeatable. I'm just wondering if that procedure  
20 can be made known, or is that a proprietary proprietary.

21 DUNLOP: There's another point here. I want to  
22 make very clear this particular point. For the particular  
23 cells that we receive in this program we were able to measure  
24 a very consistent total pre-charge, and after cycling a  
25 fairly consistent electrochemically measured precharge using

1 the procedure of a C/2 discharge into reverse, which is a  
2 destruct test.

3 If we got another size cell, or another lot of  
4 cells from G.E. today I do not believe we would get the  
5 same ratio of electrochemical to chemical pre-charge,  
6 because I made some measurements on some other cells,  
7 and they don't give you the same ratio. And the same thing  
8 applies to SAFT, by the way.

9 One of the major points that we have been making  
10 to date is that if you want to know how much pre-charge  
11 you're getting in your cells, you're going to have to use  
12 some method of determining it by both electrochemically and  
13 then by some form of chemical analysis to determine what 's  
14 remaining.

15 RAMPOL: Rampel, General Electric.

16 Jim, you mentioned before that possibly what you  
17 may do on some cells later on in orbit to determine the  
18 amount of chargeable cadmium hydroxide at the top is to  
19 continue the pre-charging sort of technique, and so on.  
20 I only want to point out, that's a good idea. But keep in  
21 mind that at that stage of the game the separator has become  
22 much drier, the recombination rate has increased, and it may  
23 be quite difficult to perform that perhaps without adding a  
24 lot of electrolyte, or something of that kind.

25 And in some cells, even adding electrolyte and

1 maintaining the close spacing that you have in the cell, it  
2 still may be somewhat impossible to fully charge up the  
3 cadmium hydroxide.

4 DUNLOP: What utilization do you expect from a  
5 G.E. plate after two years of operation?

6 RAMPEL: What plate?

7 DUNLOP: Let's take the program that we're talking  
8 about here. The plates that we have appear to have a  
9 70 percent utilization after about one or two years of opera-  
10 tion. It seems to remain fairly constant after that.

11 Is that about the number that you would expect  
12 these Intersat-4 plates to give?

13 RAMPEL: Now you're talking about the cadmium  
14 plate. Yes, I would expect that utilization to continue.

15 MAURER: I think we have a little more data  
16 here to show. Joe O'Rourke has some things he wants to show  
17 before it gets too late.

18 I would like to make one other comment, however,  
19 and that is that in doing these various manipulations of  
20 reversing and so on, we're upsetting the balance of the  
21 potassium hydroxide concentration. So we have to worry  
22 about that, too. We lose water and we change the reactivity  
23 of OH and water.

24 O'ROURKE: It's getting kind of late, and possibly  
25 the data that I have to present might confuse things. But

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1 I'll present it anyway.

2 Up until now most of the discussion centered on  
3 getting reliable, or consistent pre-charge measurements after  
4 some type of a burn-in period is first performed. The data  
5 that I have found shows that you get quite a variation in  
6 pre-charge depending upon when the actual pre-charge adjust-  
7 ment is done relative to the time from when these cells  
8 were filled with electrolyte.

9 This data was obtained on the OAO serial number  
10 34,35 battery build. These two batteries are the ones that  
11 are in the OAO satellite right outside the room there.

12 Essentially what we did -- we did this at Gulton --  
13 we measured the oxygen that was being vented during the  
14 cell build. This is with Gulton's standard pre-charge step.  
15 And we collected the gas generated, which was nearly entirely  
16 oxygen, into 1-liter graduated cylinders which were filled  
17 with water. And initially we had the tubes set in the bottom  
18 of the graduated cylinder such that the oxygen will bubble  
19 up through. And we ran into some difficulty.

20 Apparently some pressure had been formed at the  
21 top of the rubber tubing. And this created enough of an  
22 obstruction to prevent ready flow of gas out of the cell.  
23 And we're not too sure about some of the gas measurements  
24 we obtained that way.

25 After we changed the procedure and put the rubber

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1 tubing up near the top surface of the graduated cylinder  
2 while it was inverted, in such a way that there was a  
3 small air pocket there, so that when the gas came out it  
4 simply forced the level of the water down, we obtained a  
5 lot more reliable gas measurements.

6 (Slide 183.)

7 Okay. Some of these points may be hard to see.  
8 They're relatively close to the axes.

9 The x's represent the gas generated for the  
10 Gulton third electrode cell; they call it the anhydrode.  
11 And the o's represent the amount of gas that was evolved  
12 for their standard cell. And the 'x' axis is the hours  
13 of stand time from the time that the cell was filled with  
14 electrolyte until the time that the pre-charge adjustment  
15 was made.

16 And, as you can see, these points here -- I be-  
17 lieve the one closest to here was one hour stand time before  
18 the pre-charge adjustment was made. And these here were  
19 six hours soaking time before the pre-charge adjustment.

20 You can see that the gas that was evolved, the  
21 oxygen evolved, was very high and very transitory during  
22 this period, and that after about approximately ten hours or  
23 so of soaking time we got a very uniform amount of oxygen  
24 evolution.

25 Possibly what was occurring was, if the negative

1 electrode wasn't sufficient wetted, most of the gas, instead  
2 of recombining with the negative, just vented right out of  
3 the cell.

4 (Slide 184.)

5 What I did here was, I plotted the actual pre-  
6 charge negative capacity which we measured for the battery  
7 build prior to the one I just showed you. And we're seeing  
8 the same type of trend. Those cells which had the pre-  
9 charge adjustment relatively shortly after the cells were  
10 filled had higher pre-charges than those which were adjusted  
11 roughly a day after they were filled.

12 (Slide 185.)

13 This just shows a plot of the gas generated, the  
14 oxygen obtained versus the actual measured pre-charge for  
15 the 34,35 battery build. And the dotted line is the theoreti-  
16 cal 220 milliliters of gas oxygen per ampere hour of discharged  
17 negative capacity. As you can see, there's quite a bit of  
18 variation about the line but yet there is a strong correla-  
19 tion with the points also.

20 Some of the actual TN-1 measurements we made,  
21 as you can see the cells did gas. --referring to the points  
22 on the bottom, the cells did gas. And yet, as was mentioned  
23 earlier, we didn't have any electrochemical measurement  
24 obtained. This was obtained relatively shortly after the  
25 pre-charge adjustment.

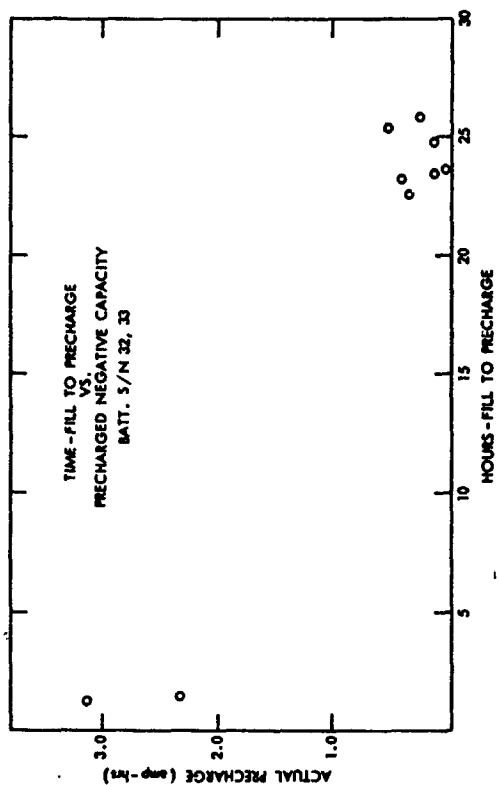


Figure 184

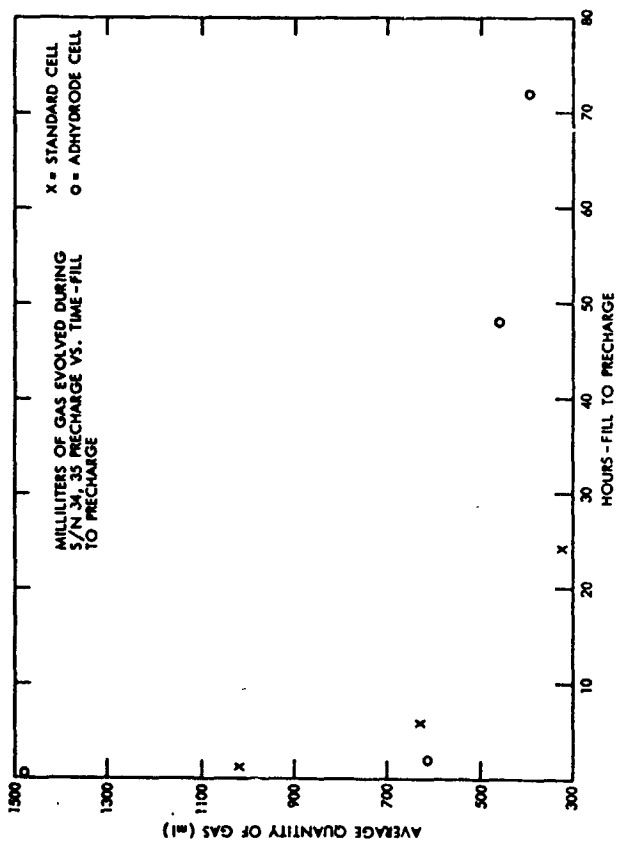


Figure 183

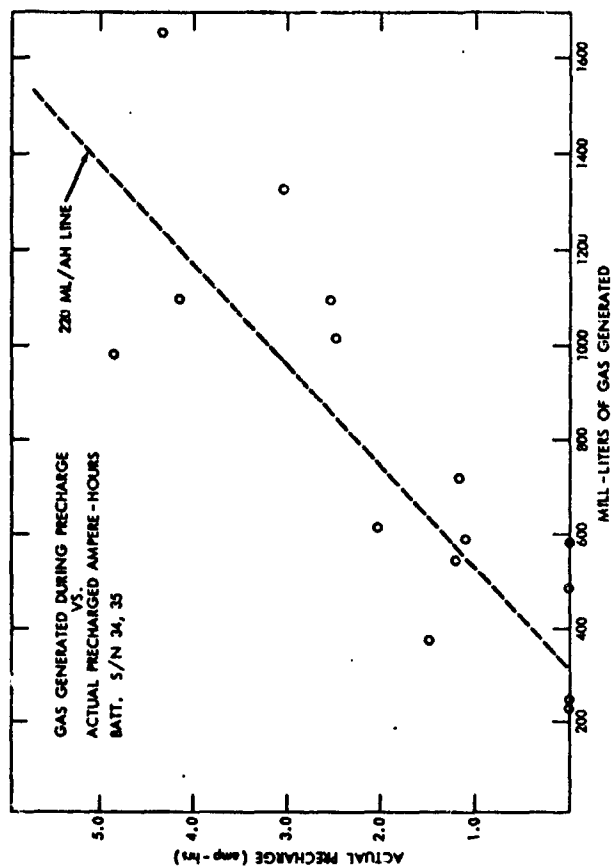


Figure 185

wb10

1                   Essentially I'm saying it wasn't much of a burn-  
2 in.

3                   That's all I have.

4                   MAURER: Any comments?

5                   BETZ: A number of things-- In my previous employ  
6 I lived through a number of these problems along with you  
7 guys. I might mention a few things.

8                   On cells very similar to the -- or pre-charged in  
9 a manner similar to OAO, that particular lot of OAO,  
10 normalizing the pre-charge we found a sigma of about six  
11 minutes and a mean of about thirty minutes. Unfortunately  
12 that -- they were all charged relatively soon after pre-  
13 charge, or after activation.

14                   Also I might mention that the reason there was  
15 a 24-hour lag was because there wasn't enough equipment  
16 to handle all the cells so that you could measure the oxygen.

17                   Secondly, at the time of this OAO build there was  
18 a different technique available for providing pre-charge.  
19 In fact I might mention it was 20-ampere-hour cells, and  
20 they're in Mariner Mars 71 out there right now. The pre-  
21 charge had an average of about 18 minutes, which would be  
22 about three ampere hours on a 20-ampere-hour cell. The  
23 standard deviation was about three minutes, as I recall,  
24 which would be comparable to Dunlop's plus or minus an  
25 ampere hour on a 15. There were relatively large samples

wb11 1 here of three to five cells per 40 for a couple of hundred  
2 cells in both cases.

3 What I wanted to say, though: the technique for  
4 measuring pre-charge will produce fairly reproducible results  
5 with reasonably large sample sizes.

6 Also, it's much better if you have some measur-  
7 able pre-charge there electrically. For instance, as I  
8 indicated recently, ATS has now achieved the ultimate: we  
9 have zero pre-charge and zero standard deviation; measurable,  
10 as you found.

11 I think the interesting point on your curve also  
12 is that there is a threshold. And in Dr. Font's data it  
13 was the same thing. That you must permit a certain amount  
14 of oxygen to escape from the initial state before you're  
15 going to measure anything. And it looks to be, in the  
16 20-ampere-hour size cells here that he had, something maybe  
17 in the order of 4 to 6 ampere hours. I think that agrees  
18 with my recollection of what the oxygen offset was, from  
19 utilizing a baseline, actually a reversible baseline.

20 But depending on the technique of pre-charge  
21 certainly there are more uniform ways available today that  
22 everybody knows about, for setting pre-charge, than what  
23 you're using on OAO. And they were available a few years  
24 ago. And I think Grumman chose not to use them.

KRAUSE: Krause, JPL. There has been a lot of

wb12, 1 concern expressed over pre-charge this afternoon. And  
2 perhaps I would like something clarified, that being where  
3 does this concern really lie? Can anyone make any general  
4 comments about failures that have occurred in application of  
5 batteries due to pre-charge problems specifically? Can  
6 anyone pinpoint that?

7 Secondly, can anyone answer if there is a type  
8 of cycling regime -- temperature, cycle life, lifetime in  
9 general -- where pre-charge is or is not a critical item?  
10 And are pre-charge techniques that are presently being used  
11 sufficient for a great number of applications, and are we  
12 getting worried over something for nothing; or is there  
13 really a problem?

14 FORD: Ford, NASA Goddard. I would like to answer  
15 that one.

16 Stan, as you, and 'most everybody here, are aware,  
17 one of the problems that led us down this path that we're  
18 currently pursuing today was back in 1968. It was involving  
19 a spacecraft battery for the OAO program.

20 We had been running cell tests, and we did observe  
21 through cell test at Goddard out of a flight lot, that  
22 after-- I remember particularly it was approximately 1300  
23 cycles in a near earth orbit at about 15 percent depth,  
24 we begin to see a cell voltage divergence on charge. This  
25 naturally did not concern us. But then as we watched it

wb13

1 progress it got worse with time, or with use.

2 To make a long story short, we resolved then that  
3 the situation that we were observing was one due to cells  
4 becoming negative limited with cycling. And I think this  
5 is attributed to -- if you look at some of the data that  
6 has been presented here and elsewhere, that when you build  
7 a cell and you look at it in the early life of it, you got  
8 good utilization in the negative, or better utilization than  
9 you do get six months downstream.

10 As you cycle the cell, the utilization in the  
11 negative falls off, and perhaps reaches some steady state  
12 value. Although I have seen data from a NASA program where  
13 certain lots of negative plates do not necessarily stabilize  
14 with cycling.

15 Now let's take the two cases.

16 If you assume that the negative plate capacity  
17 stabilizes with cycling, then all you have to worry about  
18 is having sufficient overcharge capability on Day-1 to allow  
19 that degradation in the negative electrode.

20 But then you take the second situation, which I  
21 don't have an answer to, that the negative electrode does  
22 degrade and continues to degrade; it does not show stability  
23 after a burn-in period. And I'm not sure anyone knows what  
24 a satisfactory burn-in period is.

25 We did not fail cells in the test because we had



wbl4.

1 pressure gauges, we took cell samples, and we were able to  
2 identify the problem.

3 We carried it one step further. We got involved  
4 and built another battery. But we know today we didn't  
5 resolve the problem in building that battery either, because  
6 we got those cells in-house from that lot, and we tested them  
7 approximately a year... This data is documented in the next  
8 document that I have left on the table, and part of it was  
9 reported in the 1970 Power Sources Conference.

10 We did observe, after about 4000 cycles on those  
11 flight cells for the A-2 spacecraft -- the one we've been  
12 talking about during this two-day meeting -- that one of  
13 those cells began to diverge in voltage. --divergence meaning  
14 increase. And after a period of time, approximately 1000  
15 cycles if I remember correctly, this cell began to show a  
16 gradual pressure increase with cycling. No change in test  
17 conditions, just a gradual build-up in the cell's pressure.  
18 And on a cycle-by-cycle basis this is not obvious, but on  
19 a day-by-day, and particularly a week-by-week basis, you  
20 begin to see this pressure build-up.

21 Now on that cell, by using a technique of -- we  
22 discharged the pack down, we reversed that cell; it was a  
23 C/10 rate; and removed some pre-charge, and then put the  
24 pack back on cycling. And we did indeed bring the overcharge  
25 voltage, or bring that cell back into line with the other

wbl5 1 packs.

2           Something I haven't mentioned that is pertinent  
3 to this, because I mentioned that was a group of cells out  
4 of a flight lot. Well, we have observed characteristics  
5 on that flight battery today, or earlier this summer, that  
6 lead us to believe that we have the same symptoms now in  
7 flight.

8           I hate to bring this up, because it can get quite  
9 involved, but, as most of you know, we have one cell in each  
10 battery that has a third electrode. And, incidentally, there  
11 is some correlation in the data -- looking back through the  
12 manufacturing data -- that third electrode cells going  
13 through the manufacturing process, have a certain tendency  
14 to come out with higher levels of pre-charge. And I think,  
15 without going into details, I could show you data to verify  
16 this.

17           So what we observed back during the early spring  
18 of this year, that under similar operating conditions the  
19 flight battery, the third electrode we started observing  
20 what we called an anomaly on one of the third electrodes in  
21 the flight batteries, of which there are three. We became  
22 quite concerned because we had had about two and a half  
23 years of operating time on this battery, and we saw something  
24 here that we had never really observed on ground testing -- or  
25 perhaps it's better to say we had never really looked at it

wbl6

1 in detail on ground testing.

2 What we observed is, in fact, as if the third  
3 electrode was losing sensitivity; even though we were charging,  
4 cycling, as we normally do, the third electrode on one  
5 battery just ceased to come up during charge. The other two  
6 performed as they had been in the past.

7 This went on, and we began to analyze the data.  
8 And in about another month we noticed another third elec-  
9 trode dropping out of the normal range of operation.

10 So as we looked further into this we set up a  
11 series of tests to try to find out from ground -- and this  
12 is pretty difficult to do because you have very limited  
13 data to look at. But we arranged a series of tests where  
14 we tried to analyze what the problem was.

15 Essentially we found out that if we reduced the  
16 overcharge on the battery by lowering the BVLS level con-  
17 trol, which lowers the voltage point at which you're con-  
18 trolling the battery and, consequently, lowers the re-  
19 charge, and stayed that way for two or three days, and then  
20 recycled back up to a high overcharge condition, the third  
21 electrodes all showed a very healthy response and all come  
22 back to near-normal condition.

23 We stayed there at a medium to high overcharge,  
24 in a range of 120 to 130 percent. And one of these -- possi-  
25 bly two of these third electrodes would then, over a period

wbl:f

1 of days, start decaying again and dropping out.

2 We did this on two different occasions. By that  
3 time we had done extensive investigation into the test data  
4 that we had. And we began to realize that one of the things  
5 that will kill the third electrode is the presence of  
6 hydrogen in a nickel-cadmium cell.

7 Now I can't explain all the ramifications of  
8 this, other than the fact to tell you that the test data we  
9 have shows that as you get high cell voltages and begin to  
10 generate hydrogen you can observe an apparent decrease in  
11 the sensitivity of the third electrode in the same relative  
12 oxygen environment.

13 We have seen further indications of this some  
14 time early in the program when some cells were reversed, and  
15 we noticed that on initial conditioning cycles, trying to  
16 rejuvenate the cells, that the third electrode stayed  
17 negative for quite a while, and it finally recovered over  
18 a period of two or three days. At that time we knew there  
19 was hydrogen pressure in the cell.

20 As a result of all this, we have imposed additional  
21 operating restrictions on the spacecraft. We're operating  
22 at a much reduced voltage level. We are not suffering any  
23 degradation in capacity because of this. We have reduced  
24 our recharge in the range somewhere below 110 percent; in  
25 fact, we have followed this with ground tests and demonstrated

wb18

1 that we could maintain this battery with 105 percent re-  
2 charge.

3 So I think the data that Joe O'Rourke presented  
4 yesterday adds additional information on this when he shows  
5 a chart that showed that what we call our shunt charge time  
6 had decreased by approximately about ten minutes.

7 Now the implications are simply this: We are  
8 convinced that in those flight batteries today we have  
9 considerable cell voltage divergence if we're allowed to  
10 overcharge. And when I say "overcharge" mean put in a  
11 respectable overcharge but not excessive. By maintaining a  
12 very marginal overcharge on the battery we have been able  
13 to keep the third electrodes back, and they are functioning  
14 what we consider properly.

15 But all we have to do today is to allow the battery  
16 to go to a relatively higher overcharge condition, and we  
17 will indeed see one or two of these third electrodes begin  
18 to drop out again. And it's pretty clear-cut now. This is  
19 a distinctive pattern that we have established.

20 GASTON: Gaston, Grumman. I would just like  
21 to go back to Mr. Betz' remark before, to bring it into  
22 proper perspective.

23 He somehow -- and I hope I don't misquote him: he  
24 said that there were various other pre-charge techniques  
25 available, and Grumman chose to stay with one. Well, for the

wb19

1 record, until 1968 Grumman did not know what the pre-charge  
2 procedure is; it was proprietary; and the other pre-charge  
3 techniques became, I would say about the last year or year  
4 and a half. By that time the program was qualified. And  
5 we would much rather like to stay with the one which we had  
6 before, try to control that, before we go to other techni-  
7 ques.

8 MAURER: Jim?

9 DUNLOP: Jim Dunlop, from Comsat.

10 I would like to address that question about  
11 pre-charge and why we have pre-charge.

12 It's necessary in a battery apparently to adjust  
13 the positive with respect to the negative. And the methods  
14 that are currently being used are to adjust the stated charge  
15 of the positive with respect to the negative by pre-charge.  
16 But the reason that you do it is not really-- It's not  
17 really as important how much pre-charge you put into it as  
18 how much overcharge protection you have, I believe.

19 Unfortunately, we've been talking about "pre-  
20 charge" and I think what we're really discussing in terms  
21 of the synchronous application is how much overcharge pro-  
22 tection we really have.

23 And the second point we're discussing is how to  
24 measure the amount of overcharge protection we have. And,  
25 unfortunately, as was mentioned earlier, it doesn't seem

wb20

1 that there is -- it seems that there is some concern about  
2 the best way to measure it. And what we were proposing in  
3 this, or presenting in the work that we have, is a method  
4 just to measure both the overcharge protection or the amount  
5 of protection you have before you 're going to start getting  
6 into a problem with hydrogen evolution.

7 And to do that it seems necessary to measure  
8 pre-charge.

9 KRAUSE: Krause, JPL.

10 I agree with you, Jim, and that was the point I  
11 was really driving at, that we keep talking about pre-charge  
12 and it has become apparent today that measuring pre-charge  
13 does not help you really determine what your actual over-  
14 charge protection is.

15 DUNLOP: I don't agree with that.

16 KRAUSE: Well you aren't really correlating the  
17 two, because there seems to be some inactivated species at  
18 one end that have no correlation with what is going on at  
19 the other end. And I think you've got to talk about both  
20 and not just dwell on pre-charge here.

21 I agree with you , we've got to look at both  
22 ends of this thing and not just dwell on pre-charge.

23 It seemed like there was a great concern over  
24 that one word and that one concept, and I think they are  
25 two things.

wb21

1 FORD: Maybe we've been trying to grab the  
2 donkey by its tail and we should be trying to lead him by  
3 his head.

4 LACKNER: Lackner, Canada Defense Research.

5 I agree with some of the comments that Mr. Dunlop  
6 has been saying.

7 Now he's saying we should have an effective  
8 positive to negative ratio, if I read him right. Now we  
9 have stipulated this in our satellites as being an effective  
10 1.7-to-1. We write "effective." So if there is any pre-  
11 charge it goes above that.

12 Now the way we get the effective is by matching  
13 plates. So we match the capacities of the plates, not by  
14 pre-charge.

15 So a very simple thing would be if you had a  
16 1.7 amp hour negative against a 1 amp hour positive; which  
17 you can measure in an open vat before you even put the  
18 plates together. --instead of trying to play games using  
19 pre-charge.

20 DUNLOP: Just put them together; is that what  
21 you're saying?

22 LACKNER: Yes.

23 DUNLOP: You don't do anything to them; you just  
24 put them together and seal them up?

25 LACKNER: Yes.



wb22

1                   RAMPEL: Rampel, General Electric.

2                   Ten percent pre-charge has been mentioned, and  
3 if I remained silent it could be construed that I am in  
4 agreement with that level; so --

5                   (Laughter)

6                   And I take it to mean total pre-charge, when  
7 we're saying 10 percent. Okay.

8                   Going back to Dr. Font of SAFT, with his various  
9 pre-charge levels, he summarized it and said, Well, the  
10 level of pre-charge is a compromise. I agree with that.  
11 It's necessary to have pre-charge, and I feel that we need  
12 far more than 10 percent. But by that I'm not suggesting  
13 that we go out the other end.

14                   But I'm saying here that we need more than  
15 10 percent in order to sustain problems coming from losing  
16 levels of voltage on discharge with time.

17                   DUNLOP: Guy, I think I would agree with the  
18 fact that you do need some pre-charge. And the amount that  
19 you need seems to be one of the arguments.

20                   There seem to be two arguments: the amount that  
21 you need, and how to measure it.

22                   Now taking the amount that you need: in that  
23 program that Dr. Font described, interestingly enough the  
24 levels that were used there are 2 percent, 20 percent and  
25 30 percent, approximately. You notice that the amount of

wb23

1 pre-charge in the case of the lowest one was about 8, 7 or  
2 8 ampere hours in a 72-ampere-hour cell. That's 10 percent.  
3 The second case was about 15, and the third case was about  
4 20 ampere hours of pre-charge.

5 Now we don't really know-- We do know that the  
6 pressure doesn't seem to be a factor. We also know -- I  
7 think what your comment is -- that there is an effect, an  
8 apparent effect on the measured capacity initially, ampere  
9 hour capacity of the positive plate.

10 Is that the point that you were making?

11 RAMPEL: No, sir. The point I'm making is that  
12 the cell at the 10 percent level of total pre-charge will  
13 rapidly become negative limiting with life.

14 DUNLOP: Well, Guy, the only comment I can make  
15 is that your cells that I have been testing would not have  
16 become negative limited based on the results that we  
17 presented today.

18 FORD: Are we talking about 10 percent of Day-1  
19 negative capacity, or 10 percent after burn-in?

20 DUNLOP: Floyd, as I show on this graph up here,  
21 and I'm going to repeat this many times, I guess, today,  
22 the ampere-hour capacity of those plates didn't change from  
23 Day-1 to Day-20 in terms of the total measured ampere-hour  
24 capacity of that negative plate. It was 46 ampere hours on  
25 Day-1 and it was 46 ampere hours three years later. The

wb24 -

1 total measured ampere hour capacity of those plates had not  
2 changed over that entire three-year period.

3 We didn't even see it change due to this effect  
4 that you're talking about of the soluble cadmium. We were  
5 measuring plus or minus 1 ampere hour, 46 plus or minus  
6 1 ampere hour.

7 FORD: Yes, Jim, I acknowledge that. But when  
8 we say "total", I'm concerned not with the total but with the  
9 electrochemical. And that's what I'm saying: should we base  
10 it on what the electrochemical capacity is after this burn-  
11 in period, and make it 10 percent of that? Then will we  
12 have sufficient pre-charge to prevent the problem that Guy  
13 has mentioned of fading?

14 DUNLOP: Okay, Floyd, I go back to the data that  
15 we presented today. In that cell we had a total measured  
16 electrochemical capacity, total determined. That's measured  
17 chemically and electrochemically. 46 ampere hours. We had  
18 4 ampere hours or less of cadmium charged that could not be  
19 discharged. And we're operating that cell not completely  
20 discharged; we're operating that cell between the full charge  
21 condition and the 50 percent discharged condition. We're  
22 nowhere near the point at which we might become negative  
23 limited. And we had no indication from the data that we  
24 saw that that cell would have ever become negative limited  
25 if we would have put into that cell a large -- if we had put

wb25.4

1 into that cell-- There was no reason to put into that cell  
2 more than 4 or 5 ampere hours pre-charge, based on our  
3 results to date.

4 MAURER: I would like to make a comment.

5 There seems to be a tendency among one faction  
6 here to say, Should the pre-charge be 10 percent, 20 percent  
7 or zero? I think the pre-charge should be what you need for  
8 your particular application.

9 Some of us are exposing the cells to a whole lot  
10 of overcharge, and under those conditions the amount of  
11 pre-charge can be relatively smaller. But for somebody who  
12 wants to use the cell, where he cycles it to essentially no  
13 overcharge, or even cycles it down almost upside down, then  
14 the amount of pre-charge might need to be quite a bit higher.

15 So I think we have to bear that in mind and  
16 tailor these to the application we're talking about.

17 RAMPOL: I agree, Dean.

18 And, in addition to that, pre-charge levels  
19 required may also vary between vendors' different plate  
20 properties.

21 FORD: Dean, are you saying we've finally come  
22 to the realization that we've got to design a cell for  
23 each application?

24 MAURER: Essentially.

FORD: Agreed.

wb26.111

1 STEINHAUER: Steinhauer, Hughes.

2 Does it also mean we have to develop an analytical  
3 technique for each vendor?

4 (Laughter)

5 MAURER: Well, I will comment.

6 I think you need to make measurements on each  
7 vendor with one particular technique in which you have some  
8 faith and confidence personally, and then be consistent  
9 and apply a certain set of numbers to one vendor and another  
10 set of numbers to another vendor, depending on his particu-  
11 lar properties.

12 RAMPEL: One more comment.

13 As far as the necessity for a given level of  
14 negative pre-charge, well, we did say application. But I  
15 want to emphasize that relative to temperature, high  
16 temperature, one needs a heck of a lot of negative pre-  
17 charge.

18 MAURER: Well I think we've nearly run out of  
19 questions now, so maybe we should break up and go off the  
20 record into corners and various other suitable places.

21 LACKNER: Lackner, Canada Defense Research.

22 I just have to get this title in. We talked  
23 about separators and we talked about polypropylene and  
24 wetting agents. Now in February 1970 there was a separator  
25 symposium in Columbus, Ohio, put on by the Columbus Section.

1 The Electrochemical Society, where separators were exhaustive-  
2 ly covered for nickel-cadmium, silver-zinc fuel cells. And  
3 Dr. King presented a paper on "QC Procedures and Measurements,  
4 Polypropylene Separators for Sealed Nickel-Cadmium Batteries."  
5 This is available in the standard abstracts of the Columbus  
6 Section of the Electrochemical Society meeting.

7 It covers our point of view on the separator,  
8 that a wetting agent is necessary. It goes through a whole  
9 series of tests on how we test the separator. It goes  
10 through a table which shows that air permeability does  
11 lower the pressure within the cell. And short of giving a  
12 dissertation on the separator, I will just put it on the  
13 record.

14 MAURER: Very good.

15 I'll turn the meeting over to Gerry.

16 SCOTT: May I have the last word, please?

17 In my view, the question of a satisfactory method  
18 of testing is a much simpler one than how much you need or  
19 don't need. I would hope it was one that we would have  
20 resolved a little bit more, possibly, today.

21 But, in addition to that, I would like to say that  
22 in general if one has to burn in a cell for 'x' cycles  
23 before one can get any present method of measuring pre-  
24 charge to work, then that method is suspect and not very  
25 practical, in my view. Because today in the real world of

wb28

1 purchasing cells it is generally not practical to include  
2 burn-in in acceptance testing in order to purchase cells  
3 from a supplier. And, therefore, what we need is a method  
4 which could be applied by a supplier without having to go  
5 through a burn-in procedure, in my opinion.

6 Now, whatever the user would like to do after he  
7 gets the cells is his own business. And he may be able to,  
8 indeed, make a better final determination after such a  
9 burn-in. But we still need a procedure that the supplier  
10 can use prior to that.

11 MAURER: Well I would say really that the  
12 procedure is all right, but the stated charge is really  
13 changing during that period of time.

14 SCOTT: The same question then comes back again:  
15 is it better -- can we anticipate that possibly a strictly  
16 chemical method is, in the long run, going to give us some-  
17 thing that is not so dependent upon all these conditions  
18 that we could lean on as far as some indication of what the  
19 manufacturer is doing for the purpose of quality control,  
20 and leave electrochemical measurements until sometime later  
21 on? Is that a feasible approach?

22 MAURER: Do you want to comment?

23 DUNLOP: I would like to make this comment: that  
24 I think it may require both, with the present technique,  
25 unfortunately, Dr. Scott.

wb29 - 2

1           The reason I say that is this: In that presenta-  
2           tion that Dr. Font from SAFT made, you will notice that  
3           the amount -- that it wasn't really complete because the  
4           electrochemical analysis wasn't complete at the end of the  
5           30-cycle test. But initially the data seems to agree very  
6           well.

7           If you notice, if you added together what he  
8           measured electrochemically to what he measured chemically,  
9           the result agreed with what he expected to get.

10          Did I make myself clear there?

11          In other words, it was a consistent -- the results  
12          were consistent, but that if you tried to use either, just  
13          the chemical or the electrochemical, by itself, you would have  
14          seen a variation in the results. So you have to use both.

15          And, unfortunately, in our data to date -- and  
16          let me repeat this: I think I said this once before: in the  
17          chemical analysis it is necessary for us to first completely  
18          electrochemically discharge that plate before we can make a  
19          good chemical analysis.

20          Do you understand the last point? You have to  
21          electrochemically discharge that plate.

22          SCOTT: Why?

23          DUNLOP: Because if you leave charged cadmium in  
24          there that can readily oxidize. It seems difficult to make  
25          a chemical -- not to oxidize in the process of doing the



1 chemical analysis.

2 SCOTT: You mean if you take the cell apart in  
3 an inert environment you're going to oxidize the cadmium?

4 DUNLOP: I will just say that if we on purpose  
5 charged the plate, or if you don't completely discharge it,  
6 you don't seem to get a consistent result. The only time  
7 you get a consistent result is when you completely discharge  
8 the plate electrochemically. Because the material remaining,  
9 you know, does not react either electrochemically or with  
10 oxygen at a very high rate.

11 There are other ways to discharge it, by the way,  
12 just by putting in oxygen.

13 I think there are some other people in the room  
14 that can answer that question. This has been observed not  
15 by just us but by Battelle, by SAFT, and by many people who  
16 have worked on this procedure. And it's actually documented  
17 that the best way to do it is the way I just got done  
18 describing.

19 SCOTT: If that is true, then I agree. However  
20 it would be worth while, I guess, being more sure that, for  
21 example, you could not perform a reliable chemical analysis  
22 after discharging the cells to plus 1 volt, rather than having  
23 to go through an electrochemical step.

24 FORD: I would like to make one last comment,  
25 Gerry, if I could. I've got to go on record with this,

wb3f

1 because I will have to say that with the exception of the  
2 data that Dunlop has presented I have seen no data, since  
3 we started looking at this, that tells me that the electro-  
4 chemical method of looking at ampere-hour pre-charge is  
5 satisfactory. Consequently, the only alternative we have  
6 today is just to make sure the procedure used for pre-  
7 charging or putting that charged cadmium in the cell is  
8 satisfactory and acceptable to us. That I have to go on  
9 record for.

10 And the only one that I know that is acceptable  
11 to me today is the one of a controlled oxygen venting  
12 technique.

13 DUNLOP: True; but how do you know how much to put  
14 in?

15 FORD: I have as much confidence in knowing that  
16 I did that and measured, say, 'x' number ampere hours. Now  
17 you're going to ask me how do I know what 'x' number ampere  
18 hours should be. For that I'll have to rely on experience.  
19 That's the only type of thing I have confidence in today  
20 in running a flight hardware program.

21 I cannot rely on the test technique and the  
22 numbers that result from the way it is now specified in  
23 the interim model spec in subsequent Goddard specs.

24 DUNLOP: Okay, Floyd. I agree with everything  
25 you say. But I guess I want to make just one more comment.

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1 We've presented data, SAFT has presented data,  
2 and Battelle has presented data on a procedure to measure  
3 the pre-charge. And, for some reason, you don't want to  
4 accept it.

5 Now all I'm saying is that all these procedures,  
6 all these groups that have used this particular procedure  
7 that we're describing today, have come up with a relatively  
8 consistent result. And that's the only point I'm making.  
9 Battelle, SAFT, and Comsat to date have used a procedure  
10 that we've documented, that you have access to, that says  
11 that you can measure it, and that the battery manufacturer  
12 can measure it. He can use the same procedure. They do use  
13 the same procedure. And they can measure it.

14 So it's not the fact that you-- You made a com-  
15 ment that I challenge, and the comment was that you haven't  
16 seen any data that shows you that you can measure the pre-  
17 charge. As a matter of fact, I think there has been a lot  
18 of data presented that says that you can measure the pre-  
19 charge; not just by me, but by Battelle and by SAFT today.

20 FORD: My comments still stand.

21 STEINHAFUER: Jim, if you're measuring these  
22 things, pre-charge and overcharge protection, maybe you can  
23 measure them on a relative basis. My question is, Relative  
24 to what?

25 In other words, it seems like we have to establish

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1 correlation, either chemical or, you know, electrochemical  
2 to chemical, or whatever we choose to use as the standard.

3 But I'm kind of worried, after listening to all  
4 this, and seeing members of the Spec Committee in the room,  
5 as to how long the things we heard today are going to take  
6 to get into the spec.

7 HALPERT: Let's leave it at that. That's a good  
8 question.

9 I think our questions about whether we were  
10 going to have a good meeting here this year have certainly  
11 been answered. I think we have had a good meeting. I  
12 think we look forward to next year's meeting. And I think  
13 a lot of people say they're going to do a lot of testing.

14 We have recorded in the notes all the things that  
15 they're going to do, and we're going to expect them to report  
16 on it next year.

17 We will have material, more specific data to talk  
18 about, and more specific tests, and we can identify more  
19 material.

20 I think we've gotten a lot out of this, and I'm  
21 very happy that you all attended. I want to thank all the  
22 people who have helped in chairing and in helping with the  
23 microphones, and so on.

24 I hope you all have a good trip. And we'll  
25 try and get the information to you within a month, as I

1 said.

2 Thank you all for coming.

3 (Whereupon, at 5:35 p.m., the meeting was  
4 concluded.)

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